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# Procedures Manual for Electrostatic Precipitator Evaluation

Southern Research Inst, Birmingham, Ala

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June 1977

# PROCEDURES MANUAL FOR ELECTROSTATIC PRECIPITATOR EVALUATION

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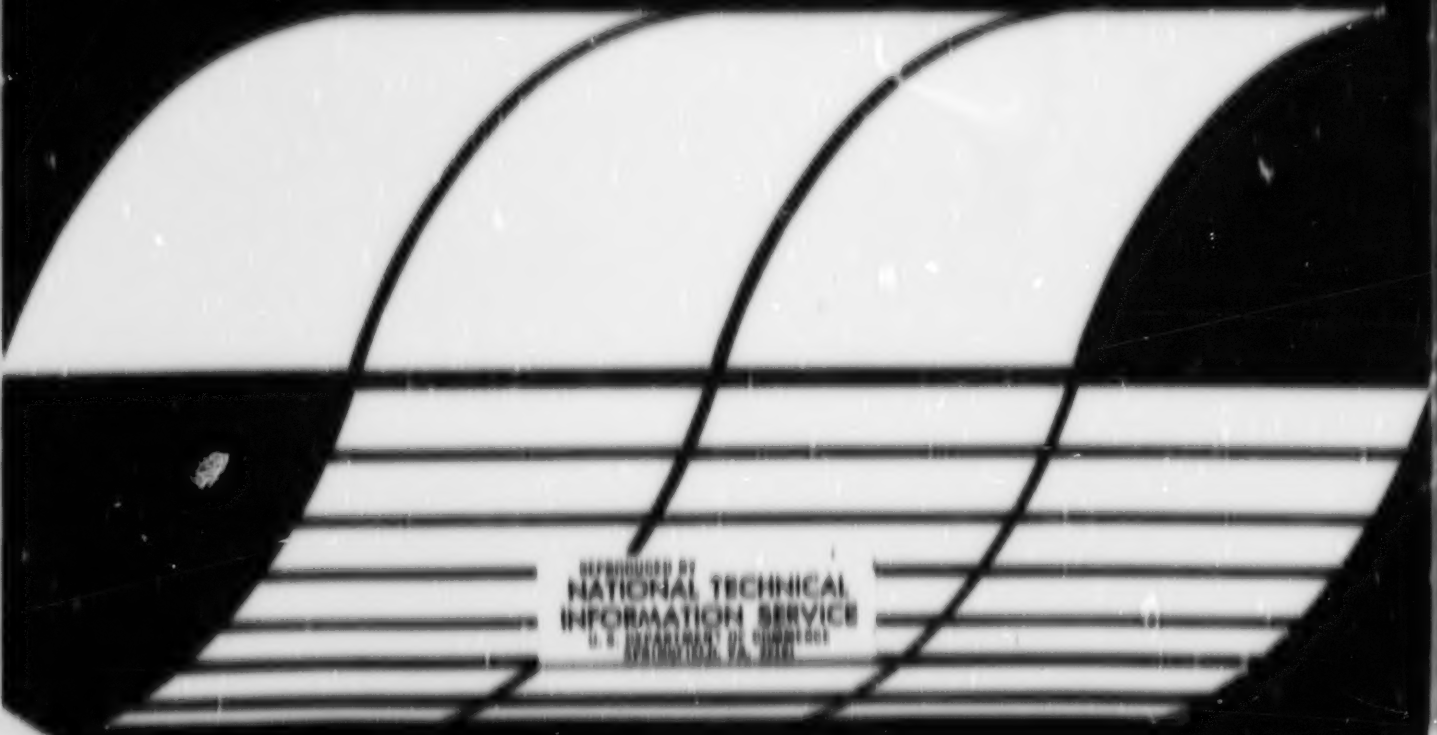
Research and Development
Program Report



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| Electrostatic Precipitators | Dust | Stationary Sources | | 11G |
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The purpose of this procedures manual is to describe methods to be used in experimentally characterizing the performance of electrostatic precipitators for pollution control. A detailed description of the mechanical and electrical characteristics of precipitators is given. Procedures are described for measuring the particle size distribution, the mass concentration of particulate matter, and the concentrations of major gaseous components of the flue gas-aerosol mixture. Procedures are also given for measuring the electrical resistivity of the dust. A concise discussion and outline is presented which describes the development of a test plan for the evaluation of a precipitator. By following this outline useful tests may be performed which range in complexity from qualitative and relatively inexpensive to rather elaborate research programs.

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We also appreciate the assistance and guidance of our Project Officer, Mr. D. Bruce Harris.

# 1. INTRODUCTION

Many different types of measurements must be made in order to accurately evaluate the performance of an electrostatic precipitator installed to remove suspended particulate matter from an industrial process stream. Among the required measurements are determinations of the compositions of the gas and suspended particles and of the particles' electrical resistivity, concentration, and size distribution. Also, the precipitator geometry and operating parameters must be recorded for proper interpretation of the measurements.

This document provides information and guidelines for use in planning and conducting tests to obtain the necessary data.

A brief description of electrostatic precipitators and various evaluation methods is provided in the remainder of this section. In Section 2 the methods of measuring precipitator operating parameters and the technical background and procedures for flue gas and particulate characterization are discussed. Section 3 describes the logic and procedure to be used in developing a test plan for the evaluation of a precipitator. The Appendices contain detailed information on the test methods, as well as a listing of the Federal Stationary Source Performance Standards and Federal Source Testing Reference Methods.

## 1.1 ELECTROSTATIC PRECIPITATOR INSTALLATIONS

### 1.1.1 Types of Electrostatic Precipitators

Two general types of electrostatic precipitators are used to control particulate emissions from stationary sources:

dry and wet electrode precipitators. Within each of these categories, precipitators may be further classified by electrode geometry. Figure 1 shows a typical parallel plate precipitator. This is by far the most common electrode configuration for large installations. Another common electrode geometry is a wire-pipe or cylindrical type.

Dry electrostatic precipitators are installed in industries with widely varying gas conditions, temperature, and pressure. In the electric utility industry there are two positions in the flue ducts for locating the collectors-- either on the hot gas or cold gas side of the air preheater

Wet electrode precipitators operate in a manner similar to hot or cold side units with the difference that a thin film of liquid flows over the collection plates to wash off the collected particulate. In some units, liquid is also sprayed into the interelectrode space to provide cooling, conditioning, or a scrubbing action. The spray is collected with the aerosol particles and provides a secondary means of wetting the plates. The operating temperatures are generally less than 65°C. Wet electrode precipitators are widely used in the metals industries.

### 1.1.2 Characteristics of Typical Precipitator Installations

Electrostatic precipitators constructed as industrial gas cleaning devices vary widely in size and configuration. The differences in size depend on the type of industrial process where they are used, the gas volume to be handled, the gas residence time, and the desired collection efficiency. Electrostatic precipitators are as large as 12 meters high,

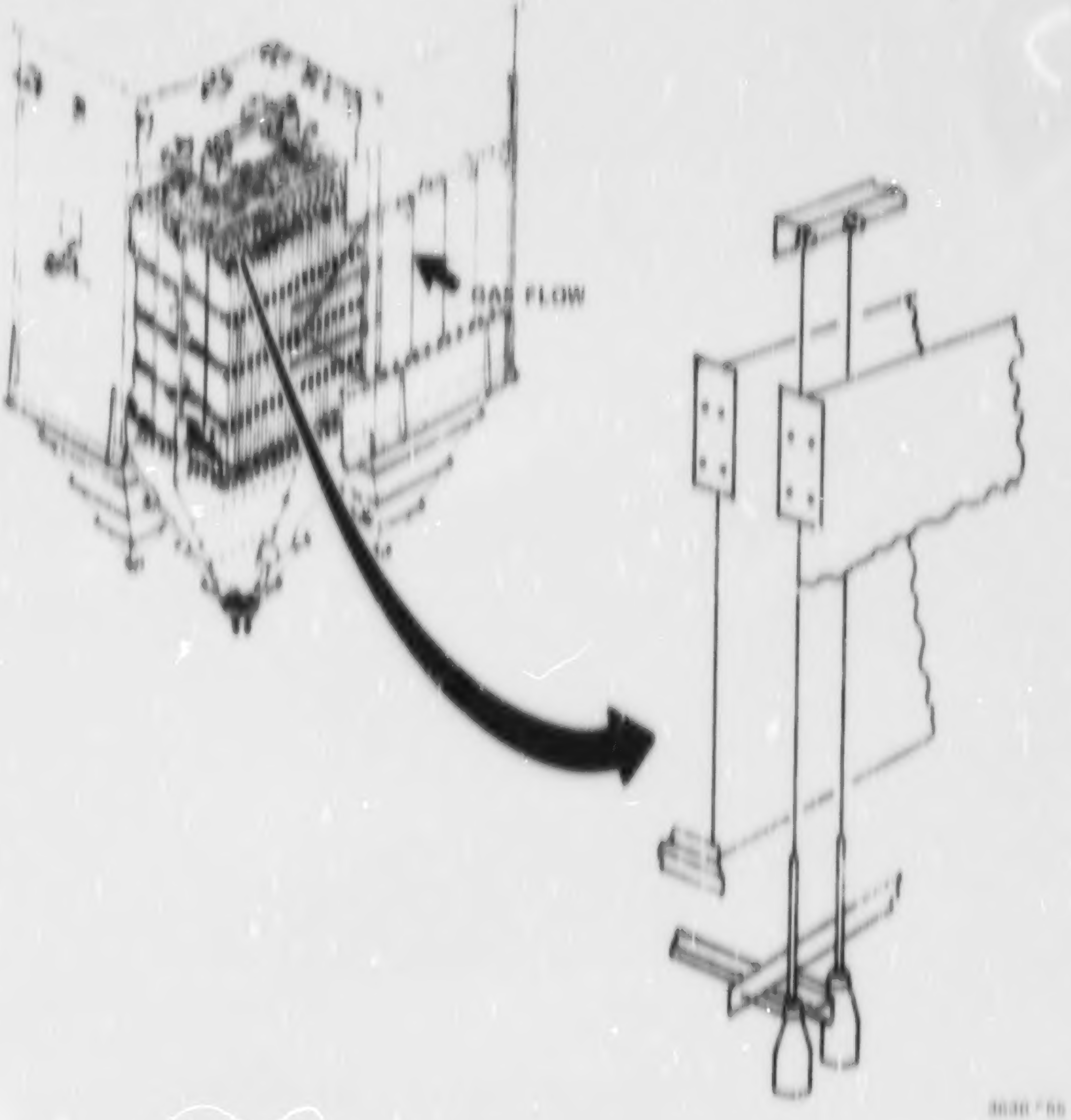

GAS FLOW
3030-55

Figure 1. Typical wire-plate electrostatic precipitator.

45 meters wide, and 30 meters long. Gas volume flow rates are as much as 1500 $m^3$/sec. For large gas volumes, however, several precipitators are usually placed in parallel. At some installations, the parallel precipitators are stacked vertically to minimize the use of ground space, especially if they are retrofitted. Figures 2 through 4 show some typical precipitator installations.

### 1.1.3 Parameters Which Govern Electrostatic Precipitator Operation

The theoretical collection efficiency, $\eta$, of a precipitator for particles of diameter D, is given by the Deutsch equation[1]

$$\eta = 1 - \exp(-w_D A/Q) \qquad (1)$$

$A$ = total effective collecting electrode area ($m^2$),
$Q$ = total gas flow rate ($m^3$/sec), and
$w_D$ = migration velocity (m/sec) of a particle with diameter D.

The migration velocity is the terminal velocity of a charged particle in the boundary layer near the collecting electrode and is largely a function of five variables:

$$w_D = w_D(j, V, Cg, T, D)$$

where
D = the particle diameter,
j = the current density in the interelectrode space,
V = the applied voltage (actually the electric field is more accurate, but is not measured directly),
Cg = the gas composition, and
T = the gas temperature.

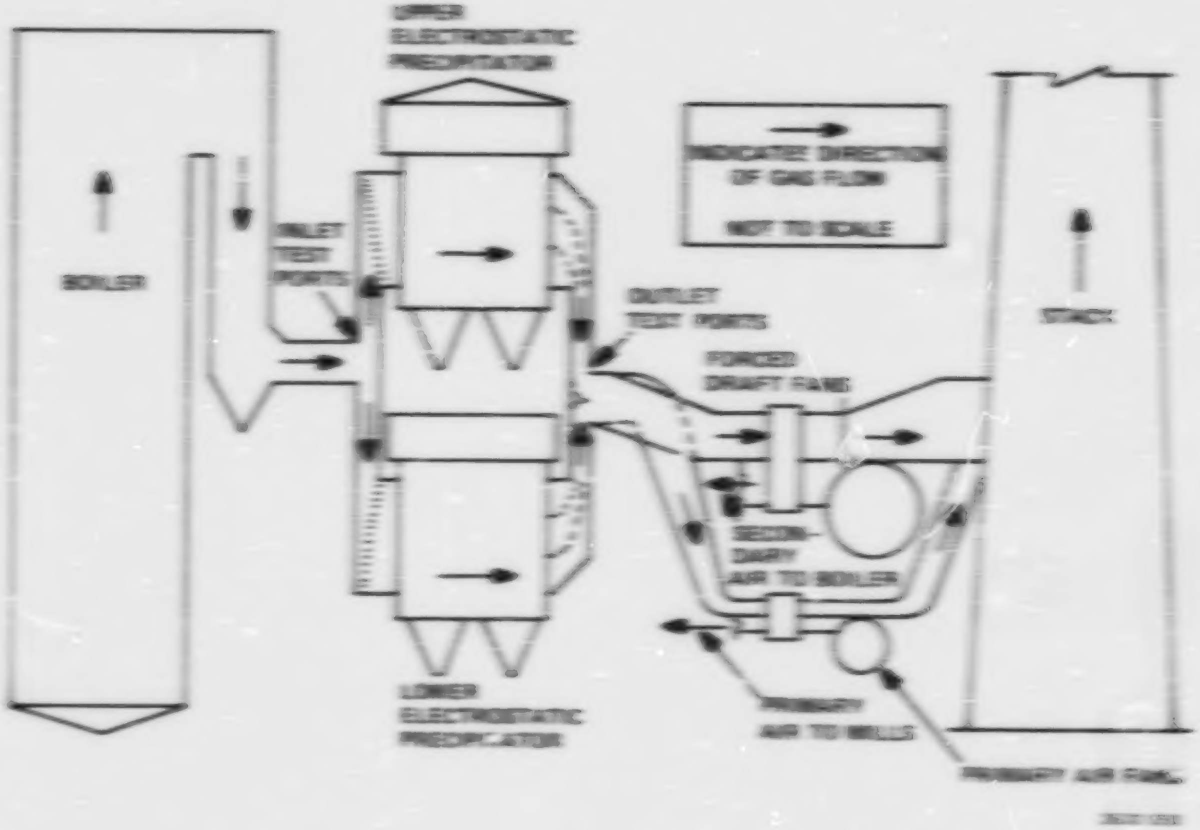

UPPER ELECTROSTATIC PRECIPITATOR
INDICATES DIRECTION OF GAS FLOW
NOT TO SCALE
BOILER
INLET TEST PORTS
OUTLET TEST PORTS
FORCED DRAFT FANS
STACK
SECONDARY AIR TO BOILER
LOWER ELECTROSTATIC PRECIPITATOR
PRIMARY AIR TO MILLS
PRIMARY AIR FANS

Figure 2. Schematic diagram of a hot side ESP installed on a coal-fired power boiler. Side view.

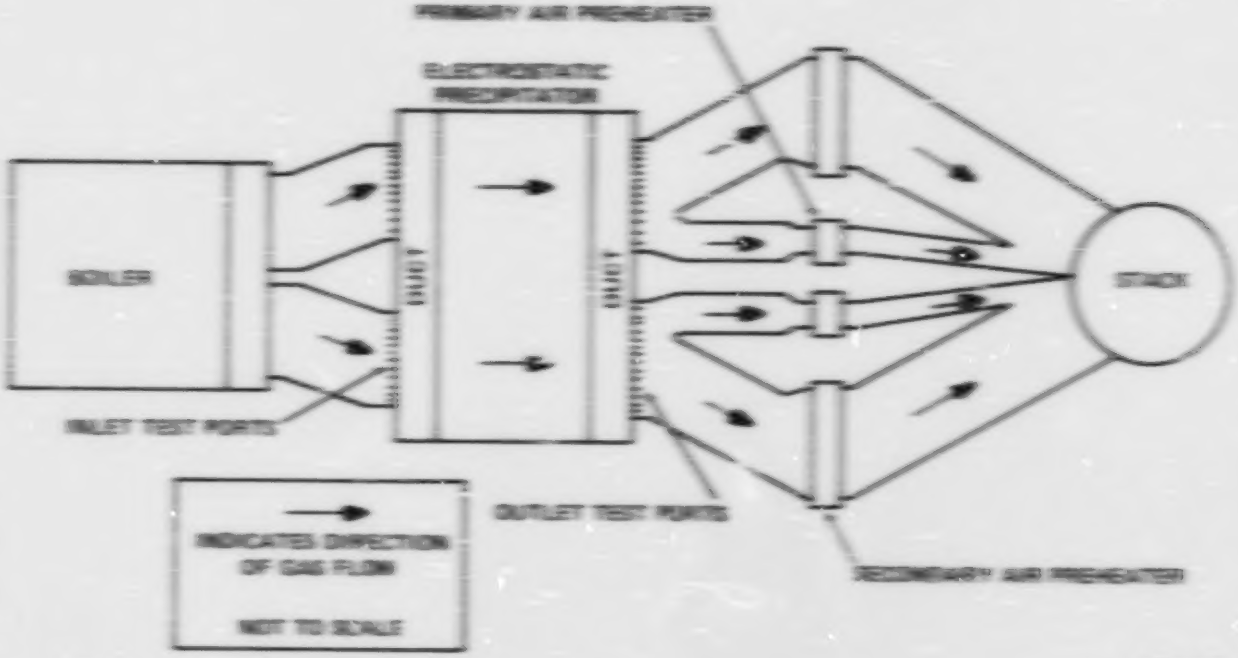

PRIMARY AIR PREHEATER
ELECTROSTATIC PRECIPITATOR
BOILER
DUCT
DUCT
STACK
INLET TEST PORTS
OUTLET TEST PORTS
SECONDARY AIR PREHEATER
INDICATES DIRECTION OF GAS FLOW
NOT TO SCALE

Figure 2. Schematic diagram of a hot side ESP installed on a coal-fired power boiler. Top view.

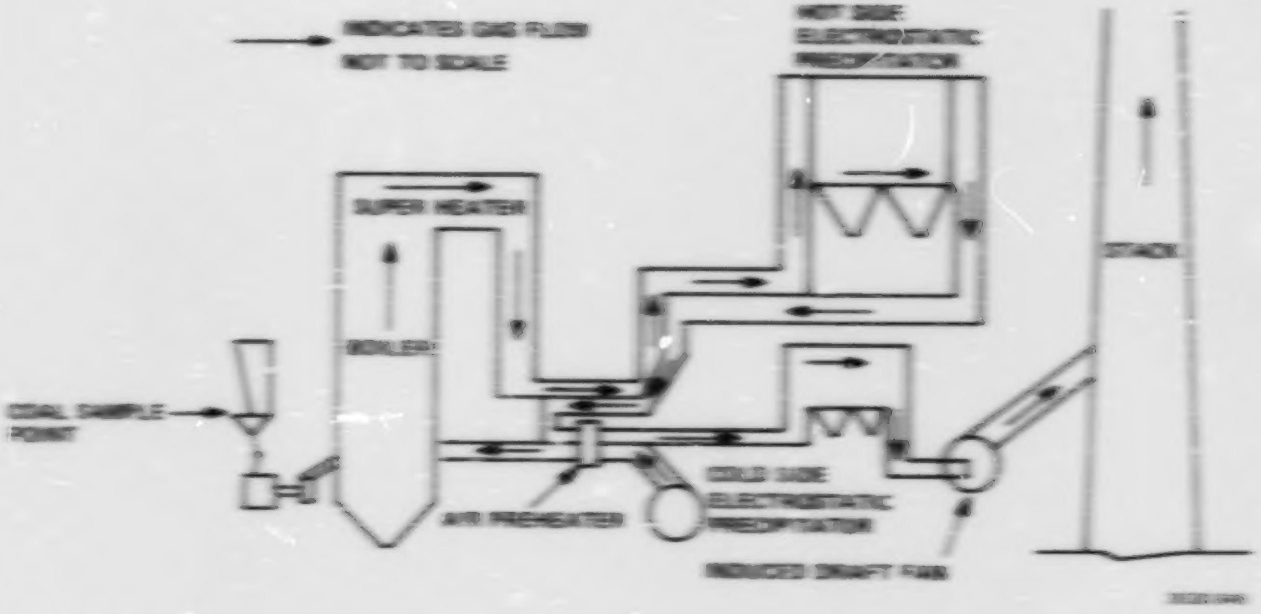

INDICATES GAS FLOW
NOT TO SCALE
HOT SIDE ELECTROSTATIC PRECIPITATOR
SUPER HEATER
BOILER
STACK
COAL SAMPLE POINT
AIR PREHEATER
COLD SIDE ELECTROSTATIC PRECIPITATOR
INDUCED DRAFT FAN

Figure 4. Schematic of an ESP system when a hot side precipitator has been retrofitted to supplement the existing cold side precipitator.

If the particle size distribution is unknown, the Deutsch equation is sometimes used with an empirical parameter, $\omega_p$, called the precipitation rate parameter, substituted for $\omega_p$. For this application, $\eta$ would represent the overall mass collection efficiency.

The six parameters A, Q, J, V, Cg, and T are the minimum number which must be measured in addition to the electrode design in order to diagnose poor precipitator performance. Of course, the actual collection efficiency can be known only after measuring the inlet and outlet mass loading concentrations, and a rigorous analysis requires a knowledge of the particle size distribution.

The quantity A/Q, measured in units of $m^2/(m^3/sec)$ is the specific collection area (SCA) used to describe the effective size of a precipitator. An SCA value of 40 $m^2/(m^3/sec)$ would be considered small for most applications and 120 $m^2/(m^3/sec)$ would be considered large. High SCA values may indicate a problem dust, a high efficiency design, or merely a conservative design.

The total collection area, A, must be obtained from the precipitator design drawings and is usually known by the plant engineer.

An approximate value of the total gas flow rate, Q, can be obtained by making a mass balance at known plant operating conditions, or by inquiring of plant personnel. During testing, however, a detailed gas velocity distribution determination by a pitot traverse across the duct or stack will give a more accurate value for total gas volume flow rate. A well defined gas flow rate distribution is necessary for proper isokinetic sampling during particulate sampling tests (i.e., the sampling velocity is equal to the gas velocity). Detailed velocity traverses and gas analyses also permit the discovery of gas leaks into or out of the precipitator.

The electric current and primary and secondary voltages are indicated by panel meters in the precipitator control room. In some instances secondary voltages must be measured using voltage dividers installed by test personnel.

Experienced personnel are able to obtain qualitative estimates of the effectiveness of particle charging, to observe resistivity problems, or to detect electrode misalignment from the experimental data listed above.

## 1.2 PARTICULATE SAMPLING FOR ELECTROSTATIC PRECIPITATOR EVALUATION

### 1.1.2 General Problems

Measurements of particle size and concentration are usually made at both the precipitator inlet and outlet to obtain an accurate characterization of the precipitator performance.

Gas velocities may vary from 15 m/sec to 30 m/sec, in ducts leading toward or away from the precipitator, to as low as 1-2 m/sec in transforms immediately upstream or downstream of the precipitator.

If samples are taken near the precipitator proper, then low gas velocities pose problems because of the difficulty of sampling isokinetically at such locations. Large diameter nozzles are required to sample at low gas velocities when the normal sampling train flow rates are used.

Particulate concentrations may range from 0.001 to 10 g/m$^3$, depending upon the type of source and the efficiency of the precipitator. Sampling times required to obtain accurate data vary approximately inversely with the dust concentration. At precipitator outlets it is not uncommon to sample for 12 hours to collect a sufficient amount of dust. Sampling times can also be discontinuous if studies are being conducted to isolate the effects of rapping reentrainment or hopper blowoff during dust removal.

At some sources, condensation can occur within the pollution control system or in the stack, and particles may grow larger and change composition, or be created, by this mechanism. Both the concentration and size distribution of the dust effect the precipitator performance.

A source of difficulty in testing a wet electrostatic precipitator is the possible presence of mists and entrained water droplets, perhaps with suspended solids, in the gas at the outlet sampling location. Also the particles can act as condensation nuclei for water droplet growth, thus causing an apparent change in the particle size distribution. Entrained water can cause filters or substrates to become soaked and erroneous measurements of the flue gas water vapor content can occur if precautions are not taken.

In many instances the duct dimensions at the sampling location can pose problems, especially if sampling from the top of a duct is required. The duct may be up to 7 meters in depth. A hoist must be constructed to handle the probes required for obtaining samples near the bottom of such a duct.

When a precipitator is sampled immediately upstream or downstream, especially when long probes are required, it is important to keep the probe from coming in contact with the corona wires as severe electrical shock can result. Unless the probes are properly grounded, sampling downstream can also result in electrical shock from the charged particles leaving the precipitator.

In a dry electrostatic precipitator the dust is collected on vertical plates which are rapped on a regular basis. Upon rapping the dust falls into hoppers and is removed from the precipitator. During the rapping cycle, however, some of the collected dust is reentrained into the gas stream and contributes significantly to the losses. This causes cyclical variations in the outlet dust concentration.

Air flow may also extend into the hoppers causing dust to be blown up and out of the hopper and back into the gas stream. This is generally localised in the lower portions of the precipitator, and a comprehensive sample traverse is necessary to include the higher dust loading at the bottom of the precipitator.

In conducting source sampling for hot side precipitator evaluations, high temperatures can cause special problems. These include difficulties in probe handling and leak sealing, filter material integrity, condensation of gaseous components upon cooling, instrument failure, etc.

In some instances, it is neither feasible nor economical to test an entire system of parallel precipitators in detail. It is reasonable to make detailed tests on a single unit and to infer the performance of untested units in the same system from electrical performance data.

### 1.2.2 Particulate Mass Measurements

Measurements of the particulate mass concentration are made by pumping the dust laden gas through a system containing a filter and a means of measuring the volume of the gas stream sampled. The total material collected on all surfaces within the system is recovered and weighed. This weight, normalized to a unit of gas volume, is the suspended particulate concentration. The samples are collected using a prescribed traversing procedure, which, in effect, yields an approximate integration of the average mass emission rate past a cross-section of the duct or stack. The velocity distribution of the gas is also measured as part of the test. The gas flow rate and velocity distribution and the particulate mass concentration are used to calculate the mass flow rate or emission rate at the point of interest. Measurements of the mass flow rate are made at both the inlet and outlet to determine the precipitator collection efficiency. Section 2.2 contains a summary of the methods for conducting mass measurements. Appendix B describes in detail how to carry out the procedures in EPA Method 5 particulate mass measurement.

### 1.2.3 Particle Sizing Techniques

Measurements of particle size distribution in industrial flue gas streams are made for several reasons. The aerosol must be characterized as completely as possible in order to assess the potential for adverse health or environmental effects; emission measurements can be useful as a process monitor; and the aerosol particle size distribution must be known in order to completely quantify the behavior of a control device. Also, particle size measurements on uncontrolled sources are useful in precipitator design.

In recent years the emphasis on pollution control has been placed on "fine" particles which are defined by the EPA as those particles having aerodynamic diameters smaller than 3 micrometers. Fine particles are more difficult to control than large particles, and because they are respirable, may constitute a greater hazard to health.

Several techniques must be applied if information on particle size over a wide range of diameters is required, or if real time data is desirable. As a general rule of thumb, most particle sizing techniques yield accurate information over approximately a factor of ten range in particle diameter. The particle size range with which this manual is concerned is 0.01 to 10 μm. Therefore, several techniques must be used.

Section 2.3 contains a summary of the instruments that are available for particle sizing, and a discussion of their applicability to specific tests. Appendices C and D contain detailed descriptions of the procedures for measurement of particle size distributions in flue gases.

### 1.2.4 Particulate Resistivity Measurements

The electrical resistivity of particulate matter, present in the effluent gas stream, is an important factor that affects the operating characteristics of an electrostatic precipitator. In a conventional single-stage dry electrostatic precipitator, the corona current must flow through a previously collected dust layer to reach the grounded collection electrode. This flow of current requires an electric field (E) in the dust layer proportional to the corona current density (j) and the particulate resistivity (ρ) as given by

$$E = j\rho \tag{2}$$

The electric field in the dust layer causes a potential difference (ΔV) across the dust layer proportional to the dust layer thickness (t),

$$\Delta V = Et \tag{3}$$

which is reflected in the operating V-I characteristic of the precipitator.

If the resistivity of the dust layer increases for a given current density, the electric field in the layer will increase proportionately. If the electric field in the interstices or on the surface of the dust layer exceeds the field strength for electrical breakdown, an electron avalanche will occur similar to that which occurs adjacent to the corona wire. This electrical breakdown acts as a limit on the allowable electrical conditions in the precipitator.

The manner in which electrical breakdown at the collection electrode limits the precipitator performance is dependent upon the value of the resistivity of the dust and the thickness of the layer. If the resistivity is in the moderately high range ($10^{11}$ ohm-cm) the breakdown will generally initiate electrical sparkover between the precipitator electrodes; whereas if the resistivity is very high ($>10^{12}$ ohm-cm), breakdown of the dust layer will occur at a voltage too low to propagate a spark across the interelectrode region. This gives rise to a condition of reverse ionization or back corona which will introduce positive ions into the interelectrode space, reducing the electrical charge on the particles.

If the particulate resistivity is too low ($<10^{7}$ ohm-cm), particles will be discharged immediately upon touching the collection electrode. If this occurs there will be no electrical forces holding the collected particles to the plate and they tend to be reentrained into the gas stream.

In view of the importance of the resistivity of the dust layer as a prime factor in limiting the performance of a precipitator, it is necessary to determine the resistivity of the material to be collected in order to perform a comprehensive evaluation of a precipitator.

Techniques of measuring particle resistivity are discussed in Section 2.4 and Appendices E and F.

# 2. TECHNICAL DISCUSSION

## 2.1 ELECTRICAL AND MECHANICAL CHARACTERIZATION OF AN ELECTROSTATIC PRECIPITATOR

In order to perform a comprehensive analysis of the performance of a precipitator it is necessary to obtain information on the design and operating parameters of the complete installation. These data are generally used, with the test results, to make comparisons with other precipitator evaluations or with performance predictions of theoretical or empirical models. Plant or process operating data should also be obtained as part of each test program and correlated with the control device performance. Plant data are routinely recorded by the plant personnel and arrangements can usually be made to obtain copies of these records or to have a member of the test crew record the data during a test.

In the remainder of Section 2.1, the nomenclature which is used to describe electrostatic precipitators is defined and a number of parameters which should ideally be measured or noted as part of a precipitator evaluation are listed. Some of the data listed are essential to a meaningful evaluation, while some may be difficult or impractical to obtain and must be sacrificed. Also, some precipitator installations may have individual peculiarities which require that additional data be taken. Although this manual is intended as a comprehensive guide, there is no substitute for intelligent and experienced judgement in the discrimination of essential from nonessential data.

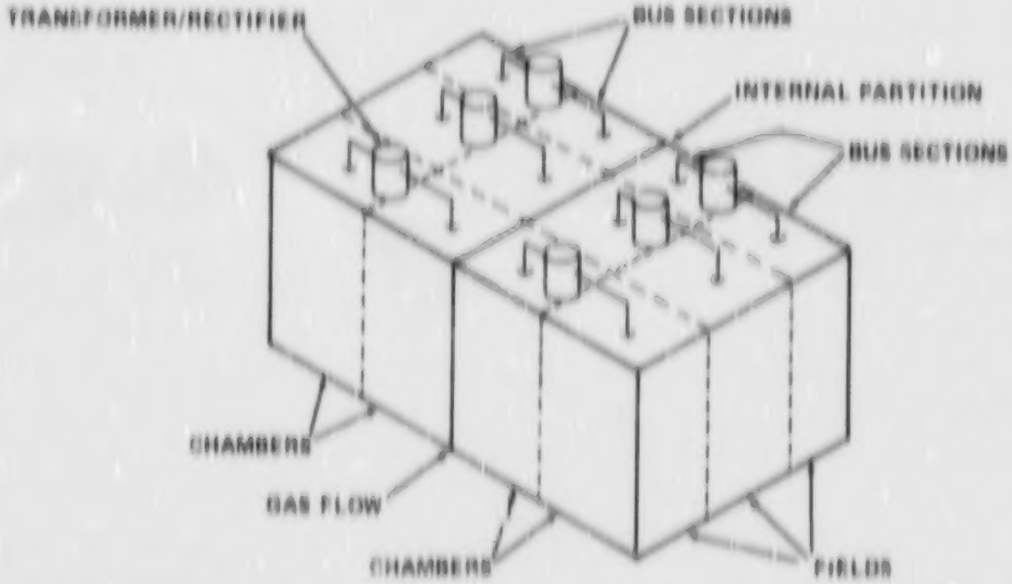
### 3.1.1 Electrical and Mechanical Design Data

The objective of a precipitator design is to combine the component parts into an effective arrangement that results in an optimum collection efficiency. Experience, efficiency requirements, and economics generally dictate the best arrangements. However, precipitator technology can be improved through the study and comparison of the electrode design, the total collecting surface area and total length of discharge electrode in one bus section, the total area of collecting surfaces cleaned by each rapper, the ratio of the area of one bus section to the total, the number of power supplies, the collecting surface area associated with one power supply, the number of fields, the total collecting surface area compared to the gas flow rate, and other parameters deemed important during the evaluation.

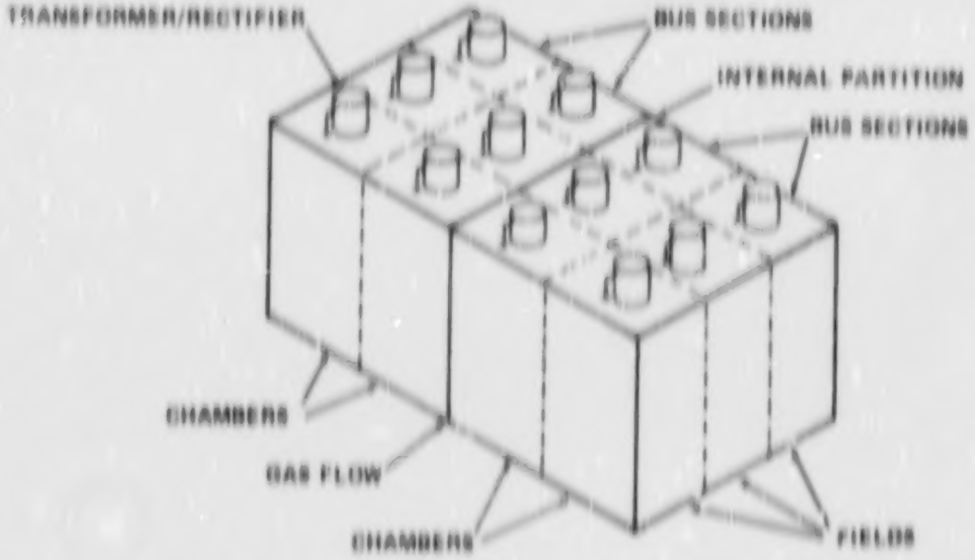
For completeness, determinations of the following items pertaining to the physical layout of the precipitator should be made during the test. This data may be obtained by observation, from plant personnel, or from manufacturers' literature.

Number of precipitators - A single precipitator is an arrangement of collecting surfaces and discharge electrodes contained within one independent housing. (See Figure 5.)

Number of chambers per precipitator - A chamber is a gas-tight longitudinal subdivision of a precipitator. A precipitator without any internal dividing wall is a single chamber precipitator. A precipitator with one dividing wall is a two-chamber precipitator, etc. (See Figure 5.)


TRANSFORMER/RECTIFIER
BUS SECTIONS
INTERNAL PARTITION
BUS SECTIONS
CHAMBERS
GAS FLOW
CHAMBERS
FIELDS

CASE I: 1 PRECIPITATOR, 2 CHAMBERS, 12 BUS SECTIONS, 6 POWER SUPPLIES, 3 FIELDS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BUS SECTIONS
INTERNAL PARTITION
BUS SECTIONS
CHAMBERS
GAS FLOW
CHAMBERS
FIELDS

CASE II: 1 PRECIPITATOR, 2 CHAMBERS, 12 BUS SECTIONS, 12 POWER SUPPLIES, 3 FIELDS

[illegible]

*Figure 5. Typical precipitator arrangements.*

*Number of fields* - A field is the physical portion of the precipitator *in the direction of gas flow* that is energized by a single power supply. A field can denote an arrangement of bus sections in parallel or series as long as this arrangement is energized by a number of parallel power supplies. (See Figure 5).

*Number of power supplies* - A power supply is a transformer /rectifier (T/R) arrangement with either a full or half wave voltage output. Usually a single power supply energizes more than one bus section.

*Number of bus sections in each field* - A bus section is the smallest portion of the precipitator which can be electrically de-energized independently. It is advantageous from a performance standpoint to have as much sectionalization as possible in a precipitator for two reasons. Localized electrical problems will not disrupt the performance of a large part of the precipitator, and since inlet sections normally operate at lower current densities than outlet sections, these should be separated electrically.

*Total number of bus sections in the precipitator* - The total number of sections in a precipitator is equal to the number of fields multiplied by the number of bus sections per field.

*Number of gas passages* - A gas passage is the volume of the precipitator between any two adjacent collecting surfaces.

*Effective precipitator dimensions* - The volume inside the precipitator through which the flue gas passes determines the effective height, width, and length of the device for the collection of dust. The effective length is equal to the total length of the collection surface in the direction of gas flow; the effective height is equal to the total height of the collecting surface measured from top to bottom; and the effective width is equal to the total number of gas passages multiplied by the center to center spacing of the collecting surfaces (ignoring the shape of the collecting surface).

Gas distribution devices - A gas distribution device is any physical plate, screen, or baffle positioned at the entrance to a precipitator to cause a change or smoothing effect in the gas flow characteristics.

### 2.1.1 Collecting Electrode System

The collecting surfaces are the individual grounded components on the surfaces of which the particulate matter is collected. The shape of the collecting electrode is designed to maximize the electric field while minimizing dust reentrainment.

Type of collection electrodes - Many shapes of collecting electrodes are used in electrostatic precipitators. For dry precipitators it is important that dust reentrainment be minimized during rapping. In well designed precipitators where gas velocities are kept low and gas flow uniformity is maintained within limits of good practice, there is little or no direct scouring of dust from the plates. All precipitators have some sort of baffle arrangement on the collection plate to minimize gas velocities near the dust surface as well as to provide stiffness to the plate. Some of these baffle-plate designs are known as Opzel plates, Rod curtain plates, Zig-Zag plates, V Plates, V-pocket plates, channel plates, offset plates, shielded plates, and tulip plates (See Figure 6).

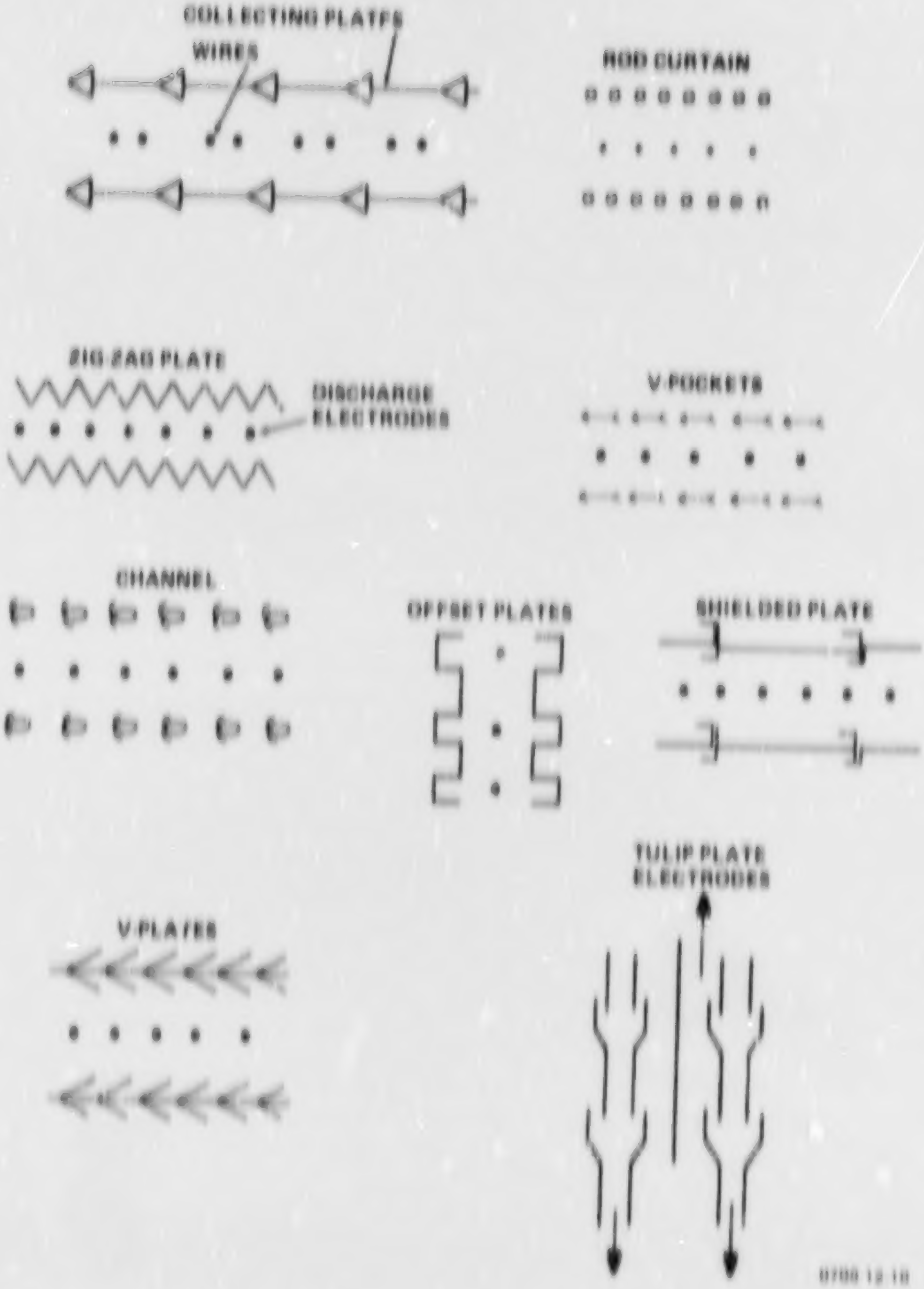

COLLECTING PLATES
WIRES
ROD CURTAIN
ZIG ZAG PLATE
DISCHARGE ELECTRODES
V-POCKETS
CHANNEL
OFFSET PLATES
SHIELDED PLATE
TULIP PLATE ELECTRODES
V-PLATES
8700 12 10

*Figure 8. Various types of collection electrodes.*

Active collecting surface area - This is the total projected area of the collecting surface exposed to the electrostatic field (effective length multiplied by the effective height and number of sides).

Plate to plate distance - This is the distance between the centers of two adjacent plates on a line perpendicular to the gas flow. The maximum current and electric field are strongly dependent upon the electrode spacing.

Aspect ratio - The collecting plate aspect ratio is the effective length divided by the effective height (L/H).

### 2.1.3 Discharge Electrode System

This system is designed in conjunction with the collecting electrode system to maximize the electric current and field strength. The discharge electrode is also referred to as the corona electrode, cathode, high voltage electrode, or corona wire. Several properties of the corona or discharge electrodes can influence precipitator efficiency and should be included in a test report.

Type of discharge electrode - The discharge electrodes are held at a high electrical potential during the precipitator operation to ionize the gas and establish electric fields for particle charging and precipitation. The electrodes may be in the form of cylindrical or square wires, barbed wire, or stamped or formed strips of metal of various shapes. Some discharge electrode geometries are shown in Figure 7.

The discharge electrode may be suspended individually from an insulating superstructure, with weights attached to the lower ends, or they may be mounted on frames, or masts. Figure 8 shows two ways that electrodes may be mounted.

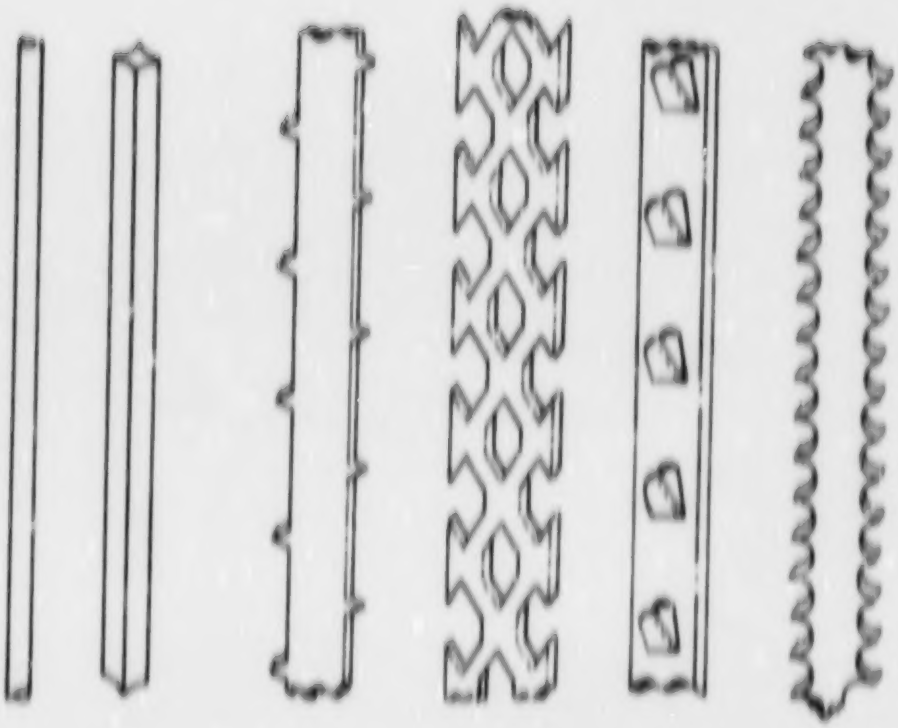


*Figure 7. Typical forms of discharge or corona electrodes.*

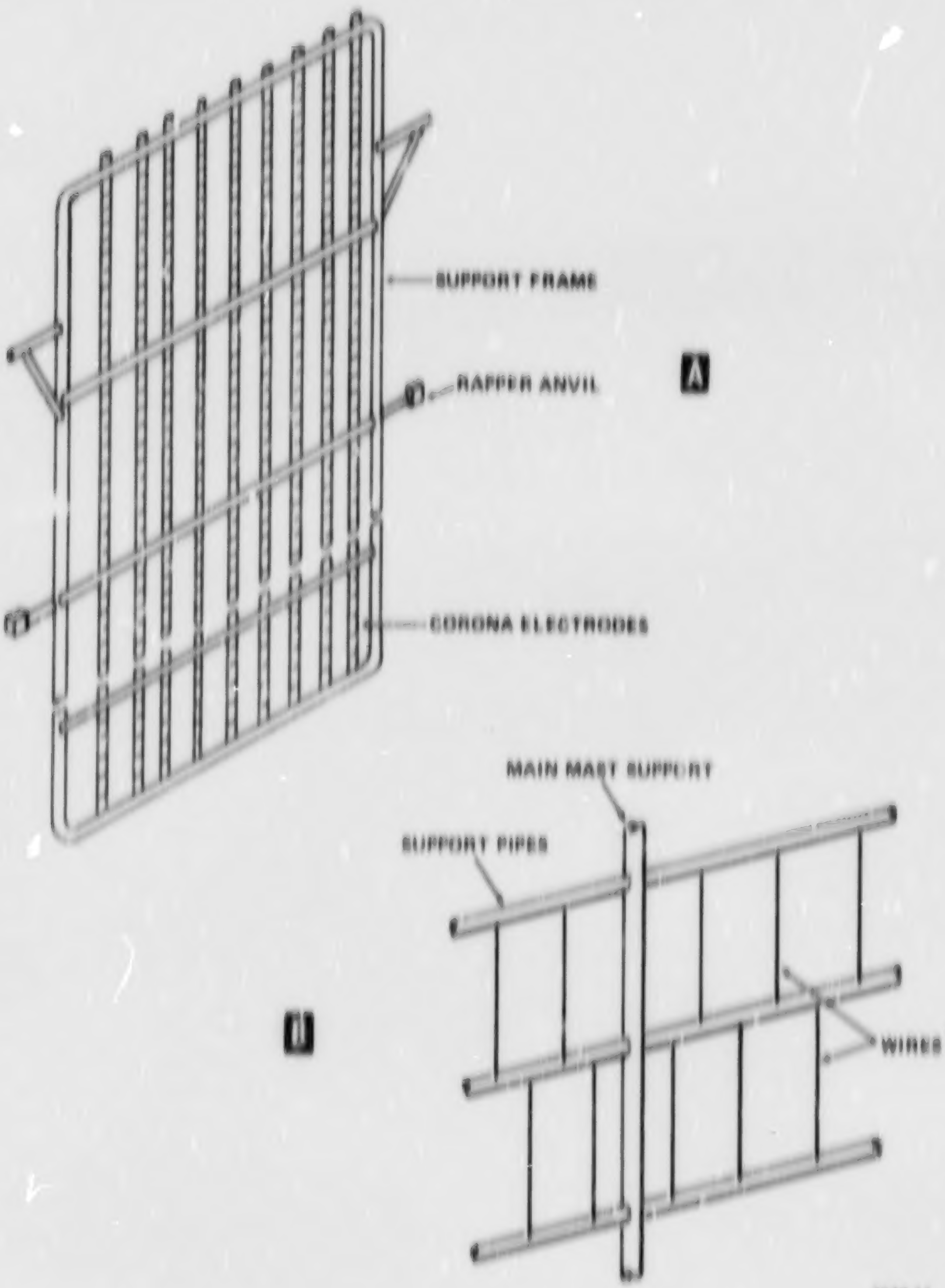

SUPPORT FRAME
RAPPER ANVIL
A
CORONA ELECTRODES
MAIN MAST SUPPORT
SUPPORT PIPES
B
WIRES

*Figure 8. Supported electrode structures.*
*A. Frame-type. B. Mast-type.*

Discharge electrode dimensions - The physical dimensions of the corona electrode should be noted. The diameter, or width of this electrode is an important factor in controlling the electric field in the interelectrode space, and the starting voltage. The effective length of the discharge electrode may also be of interest; it is equal to the height of the collection electrode.

Discharge to collecting electrode spacing - The applied voltage divided by the distance between the discharge and collecting electrodes is the average electric field.

Number of discharge electrode per gas passage - The total number of electrodes (corona wires) along the length of one gas passage may be of some interest in mathematical modeling of the precipitator.

Total number of discharge electrodes - The number of electrodes in one gas passage multiplied by the number of gas passages.

Total effective discharge electrode length - The total number of electrodes multiplied by the effective electrode length.

Spacing of discharge electrodes in the direction of gas flow - The distance between discharge electrodes in a single gas passage has a significant effect on the precipitator voltage-current characteristic.

### 2.1.4 Electric Power Supplies

Each power supply consists of four components as shown in Figure 9: a step-up transformer, a high voltage rectifier,

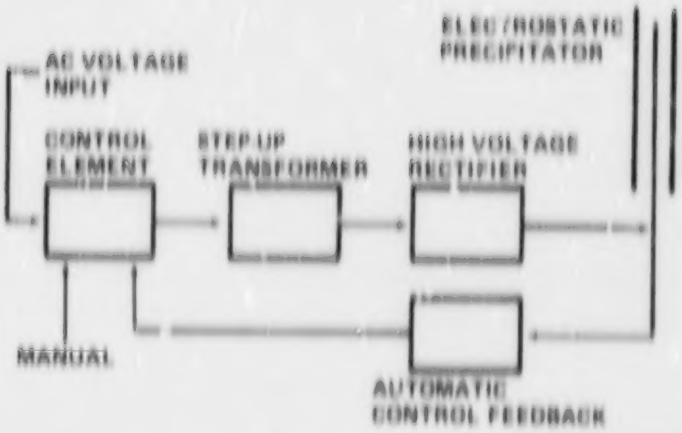

ELECTROSTATIC PRECIPITATOR
AC VOLTAGE INPUT
CONTROL ELEMENT
STEP UP TRANSFORMER
HIGH VOLTAGE RECTIFIER
MANUAL
AUTOMATIC CONTROL FEEDBACK

*Figure 8. Power supply system for modern precipitators.*

a control element, and a sensor for the control system. A step-up transformer is required because the operating voltages for modern precipitators range from about 20 to 100 kV. The high voltage rectifier is required to convert the secondary AC voltage to half or full wave voltage in order to be compatible with the electrostatic precipitator requirements.

One function of the control system is to vary the amplitude of the unfiltered voltage waveform that is applied to the discharge electrode system. Control can be applied to either the primary or secondary side in the power supply, but it is usually on the primary (low voltage) side. The control system can be operated either manually or in one of several automatic modes. Automatic controls are usually installed. A well designed automatic control system maintains the voltage level at the optimum value, even when the dust characteristics and concentration exhibit temporal fluctuations.

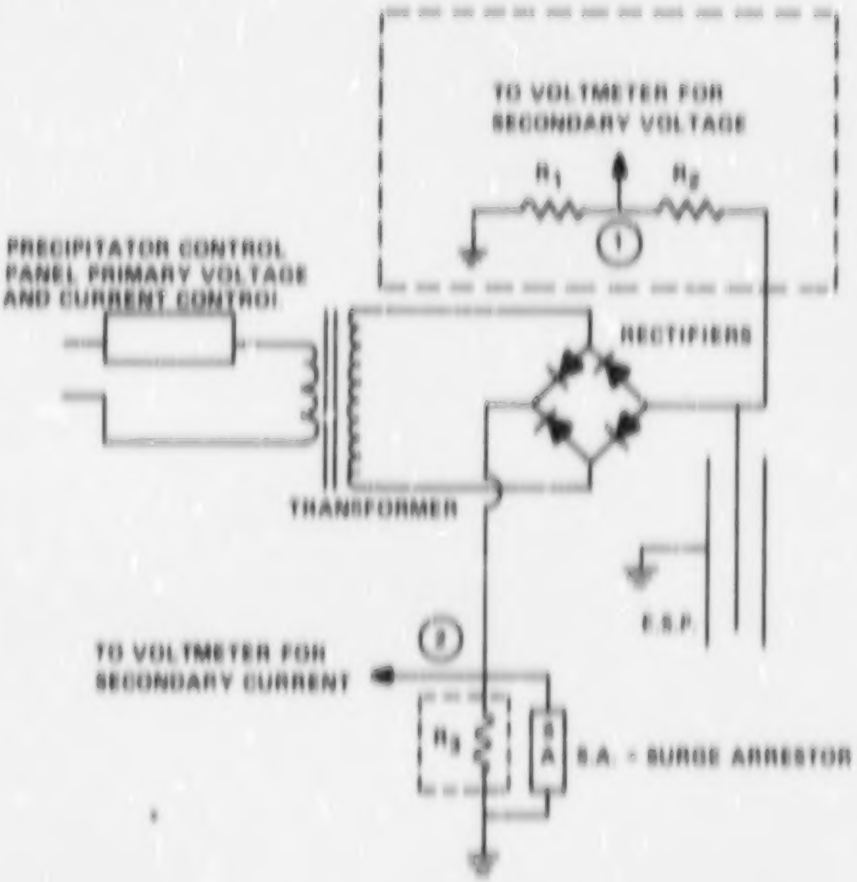
Transformers - In many precipitators, performance is limited by the maximum voltage or current ratings of the power supply transformers. The transformer ratings should be noted and reported.

Rectifiers - The rectifiers change the alternating current to a pulsating DC current, either full or half wave. In general, half wave power supplies allow a greater degree of sectionalization, although the sections are not completely independent, since control is normally associated with the transformer primary. Full wave rectification is used where higher average currents are desirable, as for example, where large dust loading or extremely fine particles lead to a large space charge which limits the maximum current.

Number of transformer - rectifier (T/R) sets - The general layout and number of T/R sets should be noted; i.e., the number per field, number per chamber, the number per precipitator. This should be shown in a schematic drawing similar to Figure 5.

Spark rate - The spark rate is the number of times per minute that electrical breakdown occurs between the corona wire and the collection electrode. A spark-rate controller establishes the applied voltage at a point where a fixed number of sparks per minute occur (typically 50-150 per corona section). The sparking rate is a function of the applied voltage for a given set of precipitator conditions. As the spark rate increases, a greater percentage of the input power is wasted in the spark current, and consequently less useful power is applied to dust collection. Continued sparking to one spot will cause erosion of the electrode, and sometimes, mechanical failure.

Voltage-Current Characteristic - Voltage-current relationships for the primary and secondary circuits of each T/R set for both clean and dirty plate conditions are very useful in interpreting precipitator behavior. Clean plate V-I data, however, must be obtained immediately after shut down after thorough plate rapping to remove excess dust or from data taken during tests occurring in the process of precipitator installation and shakedown. The dirty plate V-I measurements usually take place after particulate emission tests because it is undesirable to disturb the precipitator before the tests. When mass testing and particulate sizing measurements are being conducted, voltage and current readings should be recorded regularly for each power supply.


TO VOLTMETER FOR
SECONDARY VOLTAGE
$R_1$
$R_2$
①
PRECIPITATOR CONTROL
PANEL PRIMARY VOLTAGE
AND CURRENT CONTROL
RECTIFIERS
TRANSFORMER
E.S.P.
②
TO VOLTMETER FOR
SECONDARY CURRENT
$R_3$
S
A
S.A. = SURGE ARRESTOR

1. SECONDARY VOLTAGE = $V_1 \dfrac{R_1 + R_2}{R_1}$

2. SECONDARY CURRENT = $\dfrac{V_2}{R_3}$

*Figure 10. Voltage divider network for measuring precipitator secondary voltages and currents.*

Many precipitator control rooms have panel meters for each T/R set which show the primary and secondary voltages and current and the sparking rate. If meters are not installed on the transformer secondary, a temporary voltage divider network can be installed on the precipitator side of the recitifer network as shown in Figure 10. Typically, the resistor $R_1$ has a value of about $1 \times 10^8$ ohms and $R_2$ has a value of about $12 \times 10^3$ ohms. Because of the voltage drop across $R_1$, this resistor should be well insulated. If it is necessary to measure the secondary current, a voltmeter can be placed across resistor $R_3$ in the Surge Arrestor network. $R_3$ is typically 50 ohms or less. Some manufacturers may place a current meter with very low internal impedance across this resistor and allow all the precipitator current to pass through the meter. The resistor is installed to prevent isolating the power set should the meter be removed from the circuit. To obtain accurate data in this case, a calibrated current meter can be installed in series with this current meter. Figure 10 shows a typical precipitator T/R circuit but many individual installations may have their own peculiarities.

Since many commercial precipitators do not have secondary voltage and current meters installed, it is worthwhile for anyone involved in precipitator evaluations to build and calibrate several divider networks for field use.

### 2.1.5 Rapping Systems

Type of rappers - Differences in precipitator design also occur in the types of rappers used to remove dust collected on collection plates and discharge electrodes.

Mechanical rappers may be located within the shell of the precipitator with the rapping system consisting of hammers which are lifted by a rotating shaft until they pass over the top of the shaft and then fall under the force of gravity and strike anvils connected to the collection plates or discharge electrode frames. (See Figure 11.)

A second type of mechanical rapper is located outside of the precipitator shell with the anvils externally mounted and extending through the shell. In such a system, seals must be provided between the anvil and shell to prevent gas leakage. Rapping cycles can vary from a single impact to multiple blows for each rapping sequence and the rapping cycle can be changed by varying the frequency of rap.

Pneumatically or electromagnetically operated rappers may be of the impact or vibratory type. These rappers are connected to the discharge or collection electrode frame through the precipitator shell. The impact rapper functions by lifting a weight to a height determined by a pneumatic or electromagnetic controller and allowing the weight to fall against an anvil when the holding force is released. Vibratory rappers impart vibrations to the electrodes through rods extending into the precipitator shell. (See Figure 12).

Different rapping systems are used because of differences in dust types and manufacturers philosophy, as well as cost and maintenance considerations. The plant engineer will usually know the type of rapper and the sequence and timing of the rapping cycle.

Rapping variables - The efficiency of a precipitator is affected by the rapping interval, the rapping intensity, and the length of each rapping cycle. Rapping reentrainment

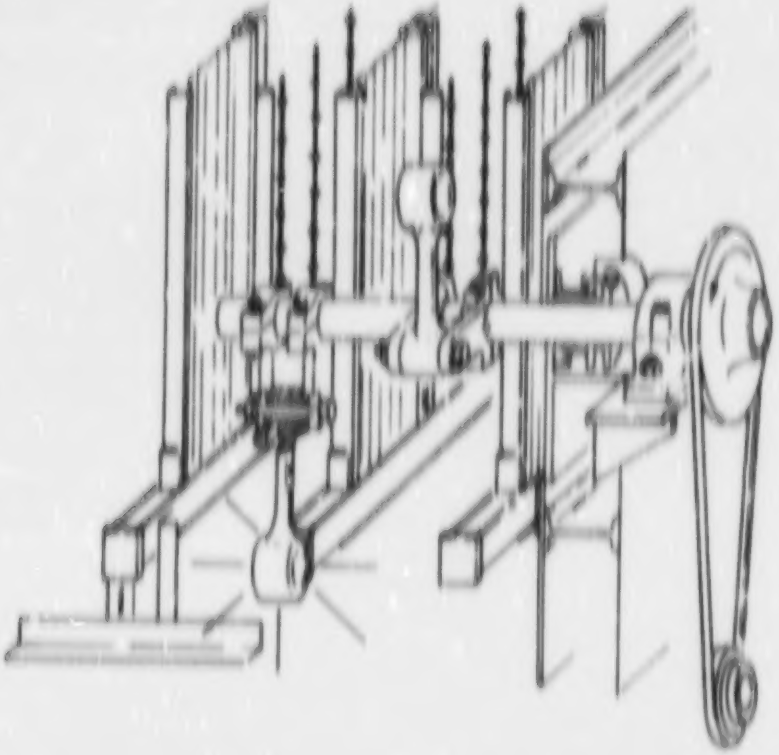


*Figure 11. Mechanical type rapper.*

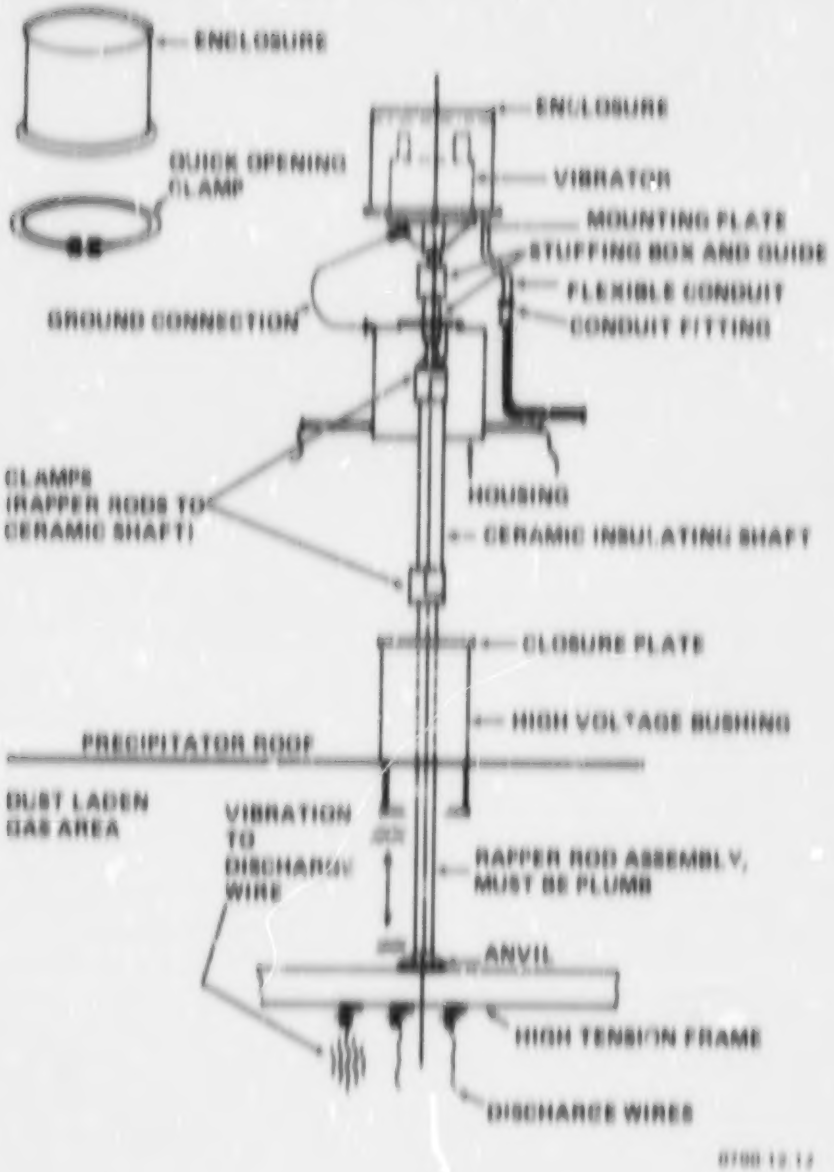

ENCLOSURE
ENCLOSURE
QUICK OPENING CLAMP
VIBRATOR
MOUNTING PLATE
STUFFING BOX AND GUIDE
FLEXIBLE CONDUIT
GROUND CONNECTION
CONDUIT FITTING
CLAMPS (RAPPER RODS TO CERAMIC SHAFT)
HOUSING
CERAMIC INSULATING SHAFT
CLOSURE PLATE
HIGH VOLTAGE BUSHING
PRECIPITATOR ROOF
DUST LADEN GAS AREA
VIBRATION TO DISCHARGE WIRE
RAPPER ROD ASSEMBLY, MUST BE PLUMB
ANVIL
HIGH TENSION FRAME
DISCHARGE WIRES
0700 12.12

*Figure 12. Typical vibratory rapper.*

contributes a significant fraction of precipitator emissions and it is usually desirable that particulate sampling at a precipitator outlet cover a time span which includes several rapping cycles. This will help ensure a more representative outlet mass loading. A different strategy would be used, of course, if the contribution of rapping to the outlet dust loading were the object of investigation.

Interval of rapping - It is desirable to know the time interval of rapping for each electrode in each field of the precipitator. Usually the upstream fields are rapped more frequently than the downstream fields because dust builds up more slowly on the outlet side.

Intensity of rapping - It is desirable to determine with what force the electrodes are being rapped within each field. Although this is an interesting quantity, it is usually impossible to obtain experimentally, except as part of elaborate research programs. Some qualitative information may be obtained from the plant engineer or manufacturing literature.

Duration of the rapping cycles - It is desirable to note the length of time of each rapping cycle for each electrode system in each field of the precipitator.

Rapper sectionalization - Precipitators are rapped field by field with the inlet fields usually being rapped more often, as noted in the report.

### 2.1.6 Dust Removal Systems

It is important during any precipitator evaluation to determine that the dust removal system is working properly and to specifications. If the system is not working properly, discussions should take place with plant personnel to see whether these problems can be fixed in a reasonable time span. If this is not the case, then testing should be delayed.

Hoppers are used to collect and store dry particulate which is removed from the electrodes. If hoppers are allowed to overflow, the collected dust will be reentrained thereby greatly reducing precipitator efficiency, or electrical short circuits may occur which disable part of the precipitator power supplies. Baffles are frequently placed in hoppers to minimize undesirable gas flow which may lead to reentrainment (hopper sweepage).

If the precipitator system is operated with internal pressures less than ambient, then air in-leakage through the hopper can cause a reentrainment of the dust from the hoppers.

Several types of systems exist for removal of dusts accumulated in hoppers. These include container removal, dry vacuum, wet vacuum, screw conveyors, and scrape bottom systems.

As much information about the dust removal system should be obtained and reported as possible.

## 2.2 MASS EMISSION MEASUREMENTS

### 2.2.1 General Discussion

As part of most electrostatic precipitator studies, the particulate collection efficiency is determined experimentally by making measurements of the mass concentration and gas flow rate at the precipitator inlet and outlet.

A number of test procedures have been developed for performing mass emission measurements on process streams. All of the "standard" sampling trains are similar and basically are composed of a nozzle, a probe, a filter, one or more devices for monitoring gas flow, and a pump. Generally pitot tubes and thermocouple assemblies are also used to measure the gas velocity and temperature. The nozzle is streamlined to minimize flow disturbances and the diameter is chosen for isokinetic sampling. The probe must be rigid so that the nozzle can be positioned accurately at the selected sampling points.

Filters of various compositions and geometries are used, although glass fiber is most common. Any filter chosen must be an efficient collector of submicron particles. A gas meter and sometimes a calibrated orifice are used to measure the gas flow rate and total volume sampled. Condensers are used to measure the water vapor content as well as for pump and meter protection. In some instances impinger bubblers and liquid traps are used and the contents analyzed after sampling for various volatile elements.

Many emissions contain substances which condense at temperatures well above ambient to form solid or liquid particles. Care must be taken that the temperature in all parts of the system upstream from the filter be kept at temperatures high enough to prevent condensation. Also, there is considerable deposition on the surfaces of the nozzles, probes, etc., and these must be thoroughly cleaned as part of normal sampling and analysis.

Three systems which are commonly used to measure mass emissions are described in the following paragraphs.

### 2.2.2 EPA-Type Particulate Sampling Train (Method 5)

Official performance testing of control devices on stationary sources in the United States must be conducted with the "EPA Method 5 Sampling Train" illustrated in Figure 13. A heated sample probe is used to transport the particulate sample to a glass fiber filter which is maintained at 160°C or more. A reverse type pitot tube is attached to the probe to insure that isokinetic sampling conditions are maintained during a traverse of the duct or stack. According to the EPA method the glass fiber filter must have a penetration value equal to or less than that of MSA 1106 BH (approximately 0.005% for a standard 0.3 µm DOP aerosol penetration test at 5 cm/sec face velocity). Gases, vapors, and any particles that penetrate the heated filter enter a series of impingers or condensers that are immersed in an ice bath. They trap the uncombined water that is present in the gas stream so that the moisture content can be determined; they also prevent the hot, humid gases from entering the gas metering system and pump.

After the traverse, the filters are dried and the probe is washed to remove and collect particulate matter from the probe walls for subsequent analysis. The EPA Method 5 requires

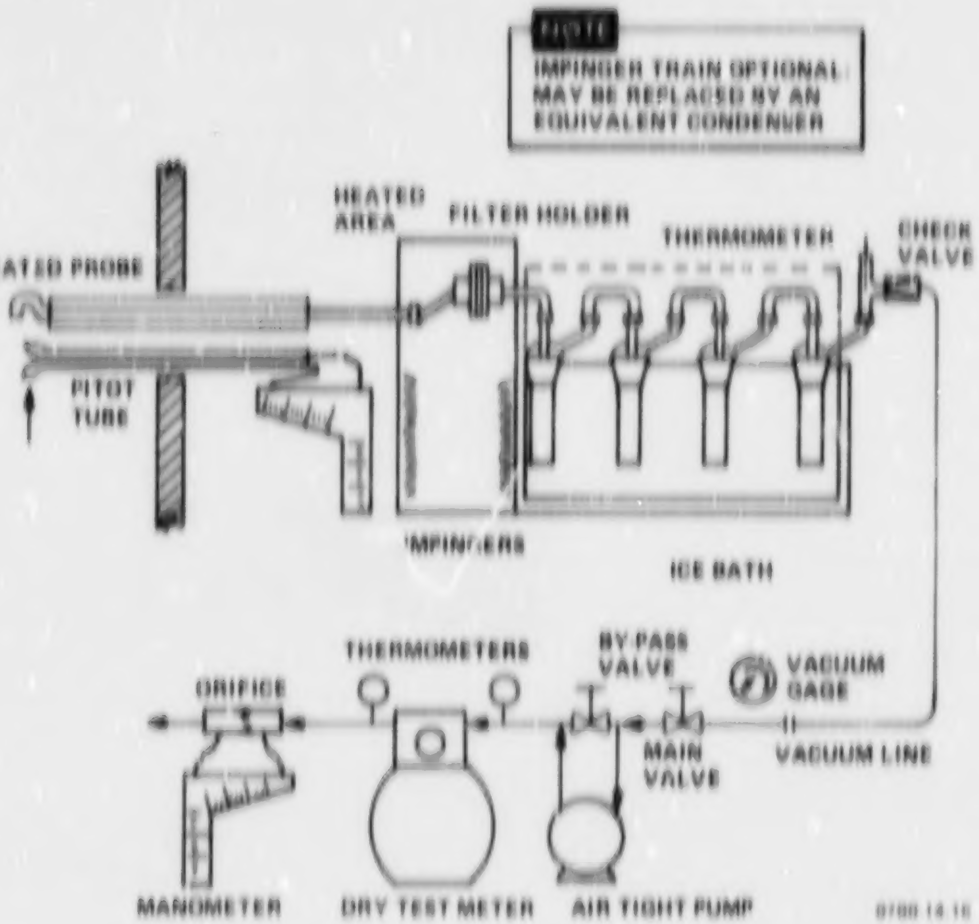

NOTE
IMPINGER TRAIN OPTIONAL:
MAY BE REPLACED BY AN
EQUIVALENT CONDENSER
HEATED AREA
FILTER HOLDER
THERMOMETER
CHECK VALVE
HEATED PROBE
PITOT TUBE
IMPINGERS
ICE BATH
THERMOMETERS
BY-PASS VALVE
VACUUM GAGE
ORIFICE
MAIN VALVE
VACUUM LINE
MANOMETER
DRY TEST METER
AIR TIGHT PUMP

*Figure 13. EPA Method 5 Particulate Sampling Train.*

that acetone be used for the probe wash. This creates a problem when the long metal probes are hot from sampling, so distilled water is often used instead of acetone. The probe wash liquid is collected and evaporated to dryness so that the amount of particulate matter removed from the probe can be weighed. This weight, and the weight of the particulate matter on the filter, and the measured gas flow are used to determine the mass emission rate of the source. Approved systems of this type are currently available in low (230-472 $cm^3$/sec) (0.5-1 $ft^3$/min.) and high (up to 170 LPS)(6 $ft^3$/min.) flow rate configurations. A modification to the EPA particulate sampling procedure (Method 17) has been proposed to allow the use of in-stack filters (See Appendix G).

### 2.2.3 ASTM-Type Particulate Sampling Train

The American Society of Testing and Materials has described a particulate sampling train which is illustrated in Figure 14. The main difference between this method and the EPA Method 5 is the use of an in-stack particulate filter. With this arrangement, a thimble-shaped filter is used to sample high mass concentrations, and a conventional, disk-shaped, filter is used for low mass concentrations. It is important to heat the filter holder to insure that the filter temperature is maintained above the dew point temperature if condensible vapors are present in the gas stream. The advantage of this system is that the particles are trapped before they enter the probe and a probe wash is not required. Also, external heating of the filter is often unnecessary. A condenser and gas cooler are still required between the probe and the gas metering system. The pitot tube, pump, and other parts of the system are similar to the EPA Method 5 Sampling Train. The thimble-filter system is not an EPA approved method but it is often used in engineering tests to evaluate the performance of a control device.

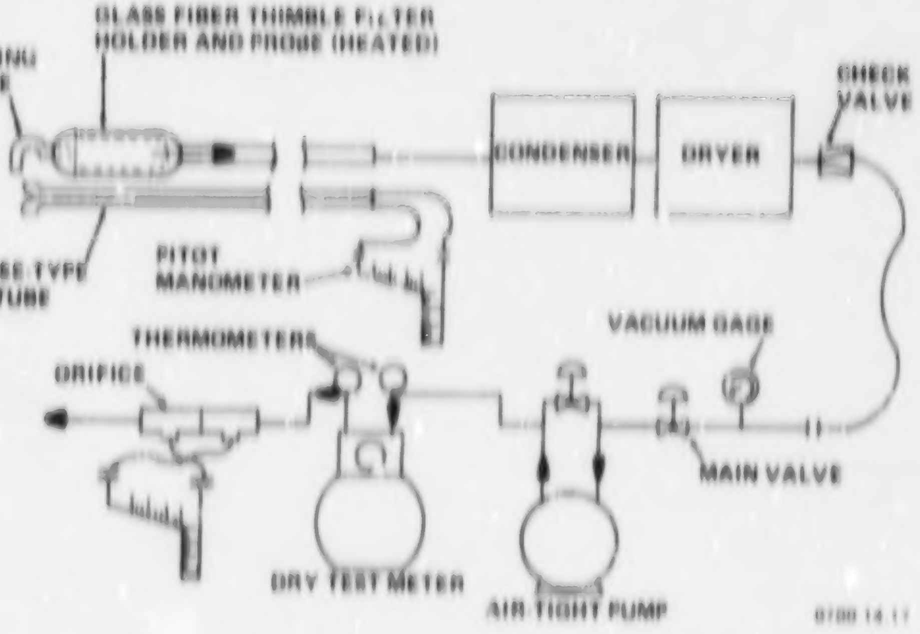

GLASS FIBER THIMBLE FILTER HOLDER AND PROBE (HEATED)
SAMPLING NOZZLE
CHECK VALVE
CONDENSER
DRYER
REVERSE-TYPE PITOT TUBE
PITOT MANOMETER
VACUUM GAGE
THERMOMETERS
ORIFICE
MAIN VALVE
DRY TEST METER
AIR-TIGHT PUMP

*Figure 14. ASTM Type Particulate Sampling Train.*

### 2.2.4 ASME Type Particulate Sampling Train

The American Society of Mechanical Engineers has described in its Power Test Codes the use of a sampling train to measure particulate emissions from industrial sources. To meet the ASME specifications the particulate sampling train must have the following parts:

A tube or nozzle for insertion into the gas stream and through which the sample is drawn.

A filter (thimble, flat disk, or bag type) for removing the particulate. For the purpose of the Power Test Code, 99.0% efficiency by weight is satisfactory.

A means of checking the equality of the velocity of the gas entering the nozzle and the velocity of the gas in the flue at the point of sampling.

A method by which the quantity of gas sampled is determined.

A pump for drawing the gas stream through the nozzle, filter, and metering device along with the necessary tubing. It is important that the temperature of the gas be above the dew point until after it has passed the filter.

This sampling procedure is not very restrictive. Both the EPA and ASTM particulate trains comply with the ASME requirements.

### 2.2.5 General Sampling Procedures

Because the EPA Method 5 is required for all compliance testing, it is the only one discussed in this section and in Appendix B where the details of performing a mass emissions test are described.

The general sampling procedure outlined herein is presented in the EPA Test Method 5 "Determination of Particulate Emissions from Stationary Sources."[7] Before sampling, however, it is necessary to determine the number of sampling points appropriate for the particular duct or stack under consideration. EPA Test Method 1 "Sample and Velocity Traverses for Stationary Sources"[7] describes the computations to determine the number of sampling points for both the velocity traverse and mass sampling traverse. The number of points will depend on the size and shape of the duct. If the velocity traverse indicates that the velocity profile in the duct is very unstable, the number of sample points should be increased to obtain a more accurate integrated mass emission rate (See Figure 15).

The use of the S-Type pitot tube, and its calibration for measuring the stack gas velocity and flow rate, is described in the EPA Test Method 2 "Determination of Stack Gas Velocity and Volumetric Flow Rate (Type S Pitot Tube)."[7] The S-Type pitot tube is used because it is less susceptible to clogging in high dust loading environments. It is also advantageous to perform a temperature traverse of the duct during the velocity traverse. This can be easily accomplished by attaching a thermocouple temperature sensor near the end

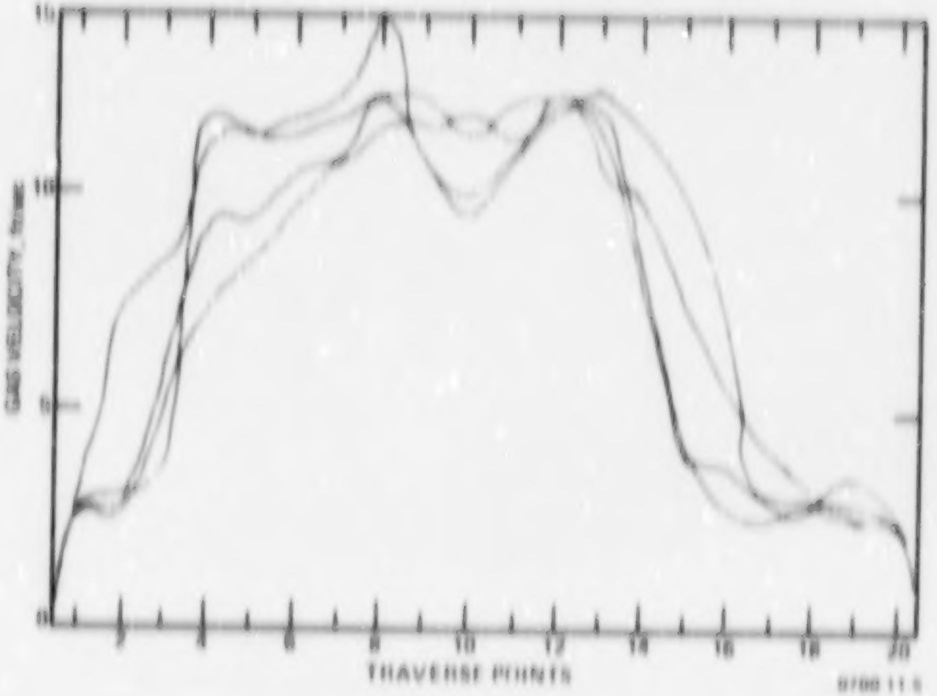


*Figure 15. Typical gas velocity distribution at the inlet to a precipitator.*

of the pitot. During actual mass sampling both the temperature and gas velocity are monitored to allow isokinetic sampling at each traverse point.

Mass sampling at electrostatic precipitators can pose problems because of the dust concentration gradients across the inlet and outlet ports. Precipitator inlet mass loadings are usually high, while outlet loadings can be up to a factor of $10^3$ smaller. Extended sampling times are required in many situations at precipitator outlets. Problems in obtaining average inlet and outlet mass loadings can also occur at sources which have cyclical operations. Outlet mass concentrations at precipitators also fluctuate because of the collection plate rapping cycles. The person preparing for a sampling procedure must take all of these variables into account when designing a test plan.

## 2.3 PARTICLE SIZE MEASUREMENT TECHNIQUES

### 2.3.1 General Discussion

Any detailed experimental program designed to evaluate an electrostatic precipitator must include measurements on the particle size distributions at the inlet and outlet. These size distributions can then be used to calculate the precipitator collection efficiency versus particle size, or "fractional efficiency curve".

Overall precipitator efficiency is strongly influenced by the inlet particle size distribution. The migration velocity, defined in Paragraph 1.1.3, is equal to the product of the electric field (E) and the particle electrical mobility. Figure 16 is a graph of electrical mobility versus particle size for a laboratory aerosol which shows a minimum value at approximately 0.1 to 0.3 μm diameter. Because of this minimum in migration velocity, fine particles, which are defined by the EPA as particles having aerodynamic diameters smaller than 3 μm, are more difficult to collect than larger particles. Unfortunately, fine particles also contribute more to visible light scattering and opacity and present a greater health hazard than do the larger particles.

At any given mass concentration, fine particle size distributions accumulate far greater total charge than large particle size distributions. The space charge associated with fine particles frequently causes sparking at relatively low voltage; this is sometimes a limiting factor in precipitator performance. Although most of the mass emitted from a particular pollution source may consist of large particles, in general, the largest number of particles is in the fine particle range. Thus, high mass collection efficiency does not always imply high number collection efficiency nor does it insure that a particular opacity standard will be met.

An ideal particle size measurement device would be located in situ and give a real time readout of particle size distributions and particle number concentration over the size range from 0.01 μm to 10 μm diameter. At the present time, however, particle size distribution measurements are made using several instruments which operate over limited size ranges and do not yield instantaneous data.

Particle sizing methods may involve instruments which are operated in-stack, or out of stack where the samples

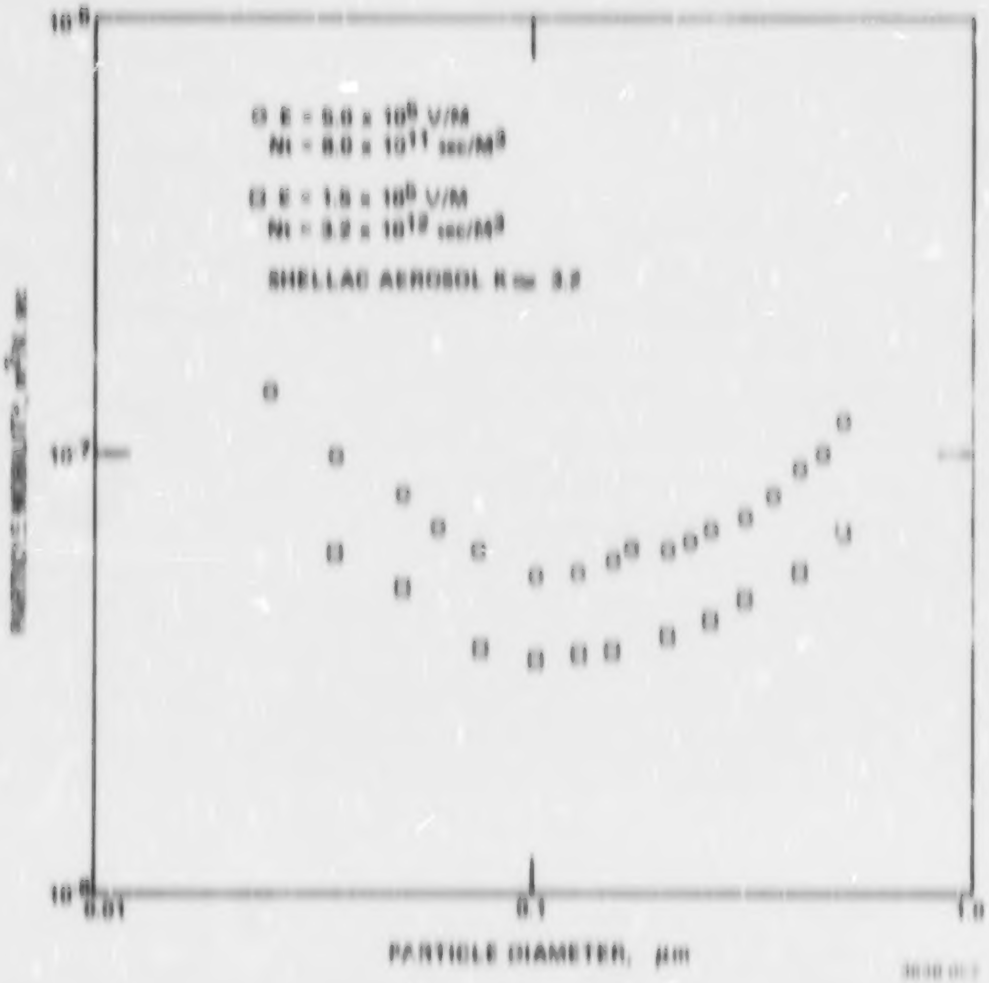


*Figure 16. Particle mobility as a function of diameter for shellac aerosol particles charged in a positive ion field (after Cochet and Trillat). K is the dielectric constant of the aerosol.*

are taken using probes. For in-stack sampling, the sample aerosol flow rate is usually adjusted to maintain near isokinetic sampling conditions in order to avoid concentration errors which result from under or oversampling large particles (dia. > 3 μm) which have too high an inertia to follow the gas flow streams in the vicinity of the sampling nozzle. Since many particulate sizing devices have size fractionation points that are flow rate dependent, the necessity for isokinetic sampling in the case of large particles can result in undesirable compromises in obtaining data - either in the number of points sampled or in the validity or precision of the data for large particles.

In general, particulate concentrations within a duct or flue are stratified to some degree with strong gradients often found for larger particles and in some cases for small particles. Such concentration gradients, which can be due to inertial effects, gravitational settling, lane to lane efficiency variations in the case of electrostatic precipitators, etc., imply that multipoint (traverse) sampling must be used.

Even the careful use of multipoint traverse techniques will not guarantee that representative data are obtained. The location of the sampling points during process changes or variations in precipitator operation can lead to significant scatter in the data. As an example, rapping losses in dry electrostatic precipitators tend to be confined to the lower portions of the gas streams, and radically different results may be obtained, depending on the magnitude of the rapping losses, and whether single point or traverse sampling is used. In addition, large variations in results from successive multipoint traverse tests can occur as a result of differences in the location of the sampling points when the precipitator plates are rapped. Similar effects will occur in other instances as a result of process variations and stratification due to settling, cyclonic flows, etc. Figure 17 illustrates temporal concentration variations due to rapping for two particle sizes at a point in space located in a duct immediately downstream of a dry electrostatic precipitator.

Choices of particulate measurement devices or methods for individual applications are dependent on the availability of suitable techniques which permit the required temporal and/or spatial resolution or integration. In many instances the properties of the particulate are subject to large changes in not only size distribution and concentration, but also in chemical composition (for example, emissions from the open hearth steel making process). Different methods or sampling devices are generally required to obtain data for long term process averages as opposed to the isolation of certain portions of the process in order to determine the cause of a particular type of emission

Interferences exist which can affect most sampling methods. Two commonly occurring problems are the condensation or vapor phase components from the gas stream and reactions of gas, liquid, or solid phase materials with various portions of the sampling systems. An example of the latter is the formation of sulfates in appreciable (several milligram) quantities on several of the commonly used glass fiber filter media by reactions involving $SO_x$ and trace constituents of the filter media. Sulfuric acid condensation in cascade impactors and in the probes used for extractive sampling is an example of the former.

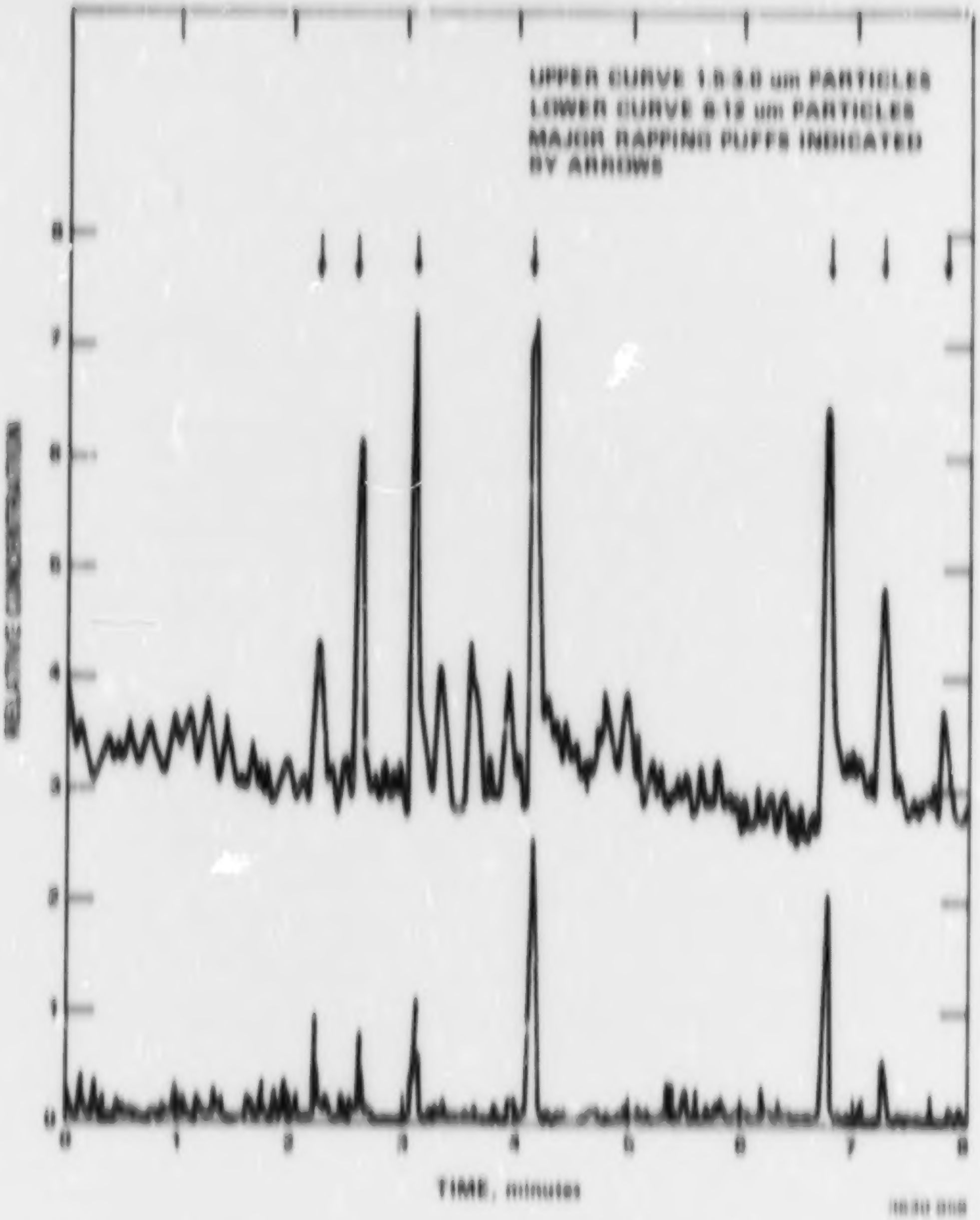


*Figure 17. Relative concentrations of particles in two size ranges between and during rapping puffs as observed at the exit of a cold side precipitator collecting fly ash from a coal fired boiler.*

If extractive sampling is used and the sample is conveyed through lengthy probes and transport lines, as is the case with several particulate sizing methods, special attention must be given toward recognition, minimization, and compensation for losses by various mechanisms in the transport lines. The degree of such losses can be quite large for certain particle sizes.

In this section, individual particulate sampling systems and procedures are discussed. These are categorized according to the physical mechanism that is used to obtain the data: inertial, optical, electrical, or diffusional.

### 2.1.2 Inertial Particle Sizing Devices

Two devices which fall into the inertial sizing category are impactors and cyclones. In both of these devices, the aerosol stream is constrained to follow a path of such curvature that the particles tend to move radially outward toward a collection surface because of their inertia. Subsequent analysis of the particle size distribution may be made by gravimetric means, quantitative chemical analysis, or microscopic inspection.

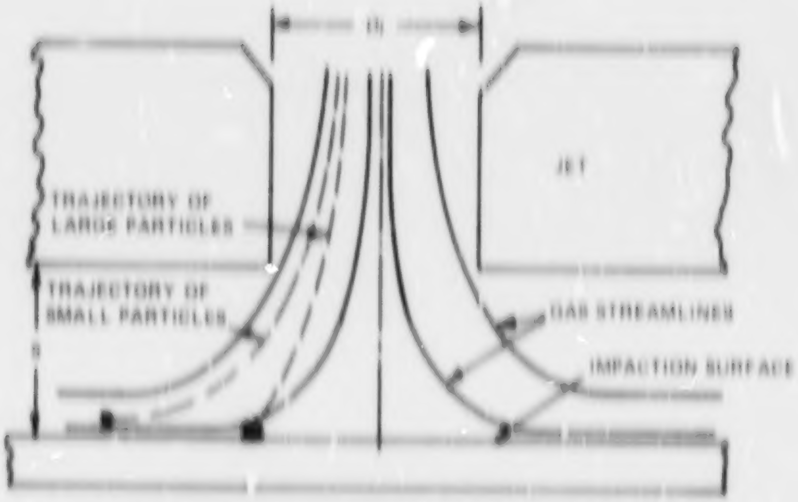
Particle size distribution measurements related to precipitator evaluation have largely been made using cascade impactors, which are effective in the size range from 0.3 to 20 um diameter; although, in some cases, hybrid cyclone-impactor units, or cyclones have also been used. The particle size distributions are normally calculated from experimental data by relating the mass collected on various stages to the theoretical or calibrated size cutpoints associated with those stage geometries.

Cascade impactors - Because of its compact arrangement and mechanical simplicity, the cascade impactor has gained wide acceptance as a practical means of making particle size measurements in flue gases. In most cases, the impactors can be inserted directly into the duct or flue, eliminating many condensation and sample loss problems which occur when probes are used for extractive sampling.

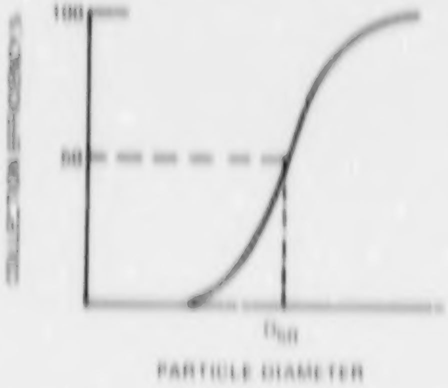
Figure 18 is a schematic which illustrates the principle of particle collection which is common to all cascade impactors. The sample aerosol is constrained to pass through a slit or circular hole to form a jet which is directed toward an impaction surface. Particles which have lower momentum will follow the air stream to lower stages where the jet velocities are progressively higher. For each stage there is a characteristic particle size which theoretically has a 50% probability of striking the collection surface. This particle size, or $D_{50}$, is called the effective cut size for that stage. The number of holes or jets on any one stage ranges from one to several hundred depending on the desired jet velocity and total volumetric flow rate. The number of jet stages in an impactor ranges from one to about twenty for various impactor geometries reported in the literature. Most commercially available impactors have between five and ten stages.

Parameters which determine the collection efficiency for a particular geometry are the gas viscosity, the particle density, the jet diameter or width, the jet-to-plate spacing, and the velocity of the air jet.

Most modern impactor designs are based on the semi-empirical theory of Ranz and Wong.[9] More comprehensive theories


$D_j$
JET
TRAJECTORY OF LARGE PARTICLES
TRAJECTORY OF SMALL PARTICLES
S
GAS STREAMLINES
IMPACTION SURFACE

A. TYPICAL IMPACTOR JET AND COLLECTION PLATE



B. GENERALIZED STAGE COLLECTION EFFICIENCY CURVE

[illegible]

*Figure 18. Operation principle and typical performance for a cascade impactor.*

have been developed by Davies and Aylward[5] and by Marple.[6] In practice, deviations from ideal behavior in actual impactors dictate that they be calibrated experimentally.

A large number of experimental studies have been published on cascade impactor design and performance in the laboratory environment. Most of these have been reviewed in the dissertations of Marple[6] and Rao.[7] Recently, Cushing et al[8] have presented calibration data on several commercially available cascade impactors. Figures 19, 20, 21, 22, and 23 show schematics of the commercial impactor designs which are commonly used in source testing. Table 1 gives a listing of the manufacturers, and some operational information for stack sampling. The details of cascade impactor applications are discussed in Appendix C.

It is usually impractical to use the same impactor at the inlet and outlet of a precipitator when making fractional efficiency measurements because of the large difference in particulate loading. For example, if a sampling time of thirty minutes is adequate at the inlet, for the same impactor operating conditions and the same amount of sample collected, approximately 3000 minutes sampling time would be required at the outlet (a collection efficiency of 99% is assumed). Although impactor flow rates can be varied, they cannot be adjusted enough to compensate for this difference in particulate loading without creating other problems. Extremely high sampling rates result in particle bounce and in scouring of impacted particles from the lower stages of the impactor where the jet velocities become excessively high. Short sampling times may result in atypical samples being obtained as a result of momentary fluctuations in the particle concentration or size distribution within the duct. Normally, a low flow rate impactor is used at

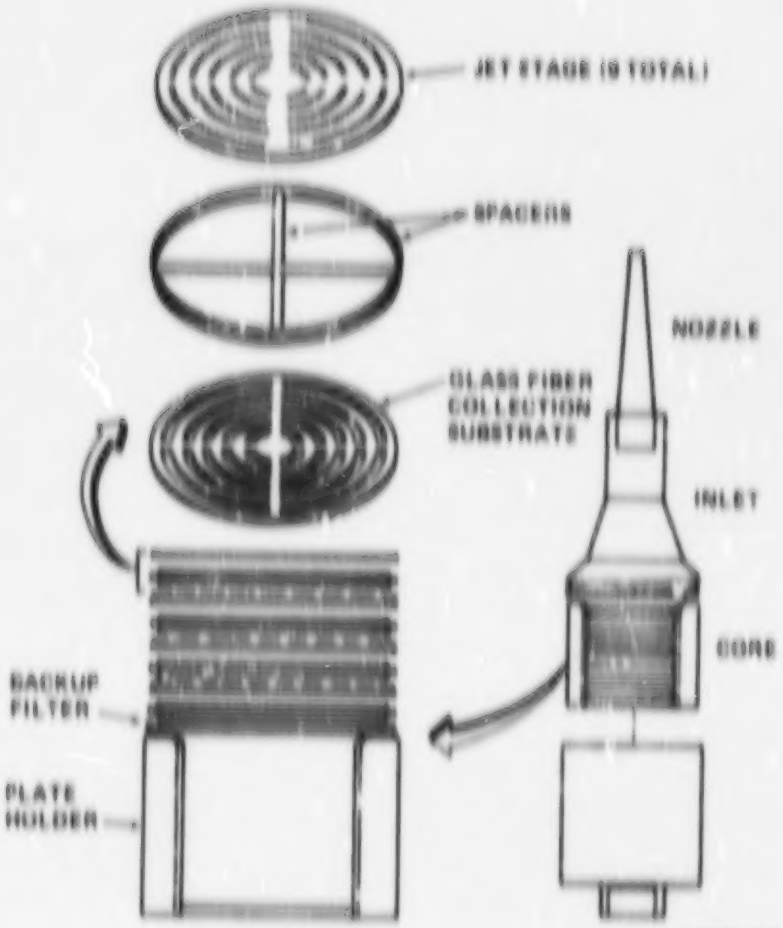

JET STAGE (9 TOTAL)
SPACERS
NOZZLE
GLASS FIBER COLLECTION SUBSTRATE
INLET
CORE
BACKUP FILTER
PLATE HOLDER

*Figure 19. Andersen Mark III Stack Sampler.*

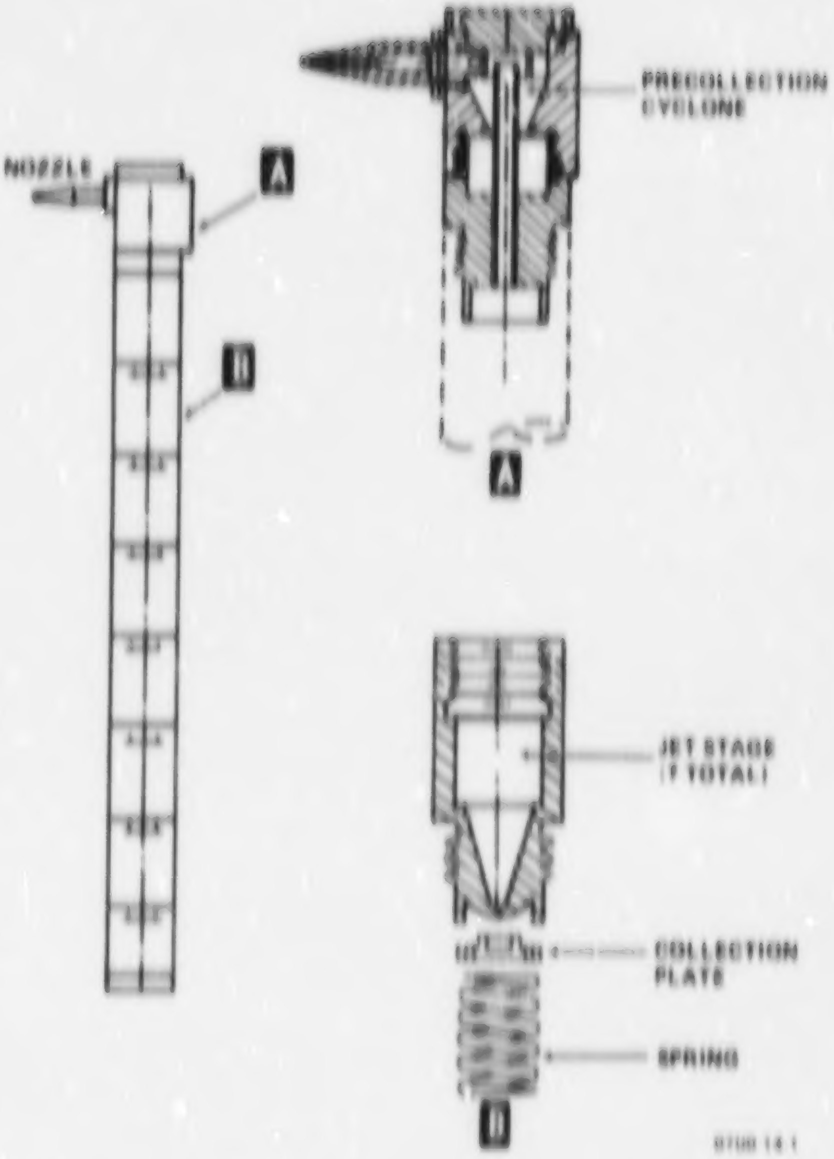

PRECOLLECTION CYCLONE
NOZZLE
A
B
A
JET STAGE (7 TOTAL)
COLLECTION PLATE
SPRING
B

*Figure 20. Modified Brink Model BMS-11 Cascade Impactor.*

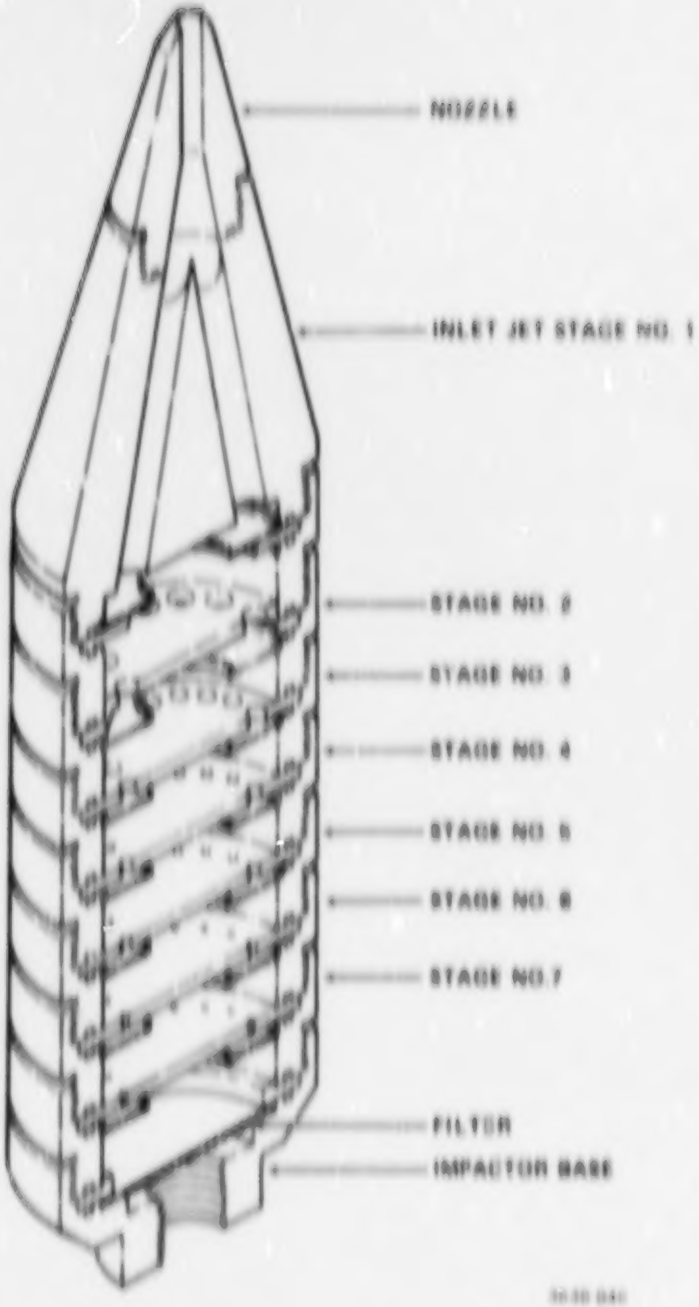

NOZZLE
INLET JET STAGE NO. 1
STAGE NO. 2
STAGE NO. 3
STAGE NO. 4
STAGE NO. 5
STAGE NO. 6
STAGE NO.7
FILTER
IMPACTOR BASE

*Figure 21. MRI Model 1502 Inertial Cascade Impactor.*

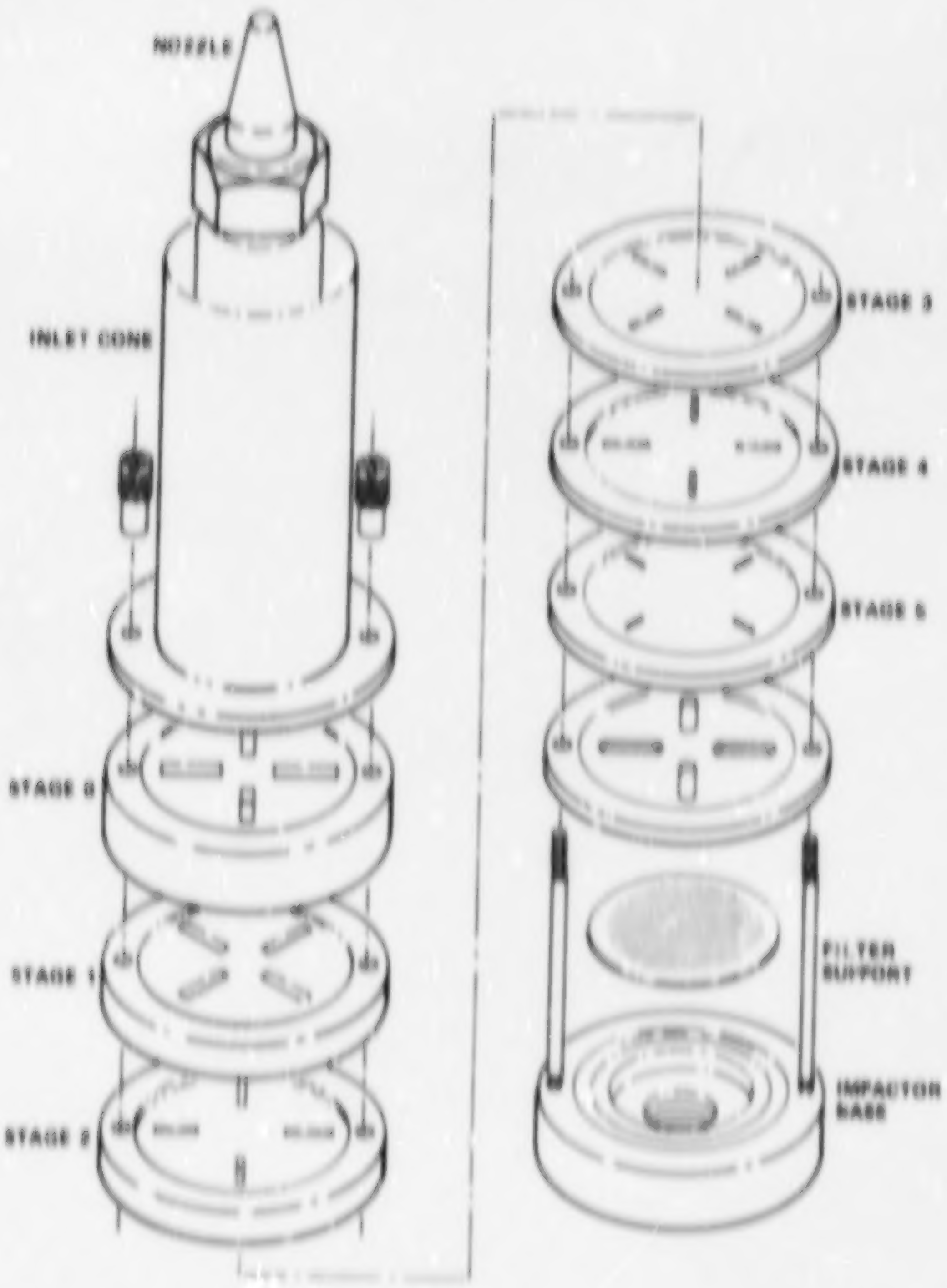

NOZZLE
INLET CONE
STAGE 0
STAGE 1
STAGE 2
STAGE 3
STAGE 4
STAGE 5
FILTER SUPPORT
IMPACTOR BASE

*Figure 22. Sierra Model 226 Cascade Impactor.*

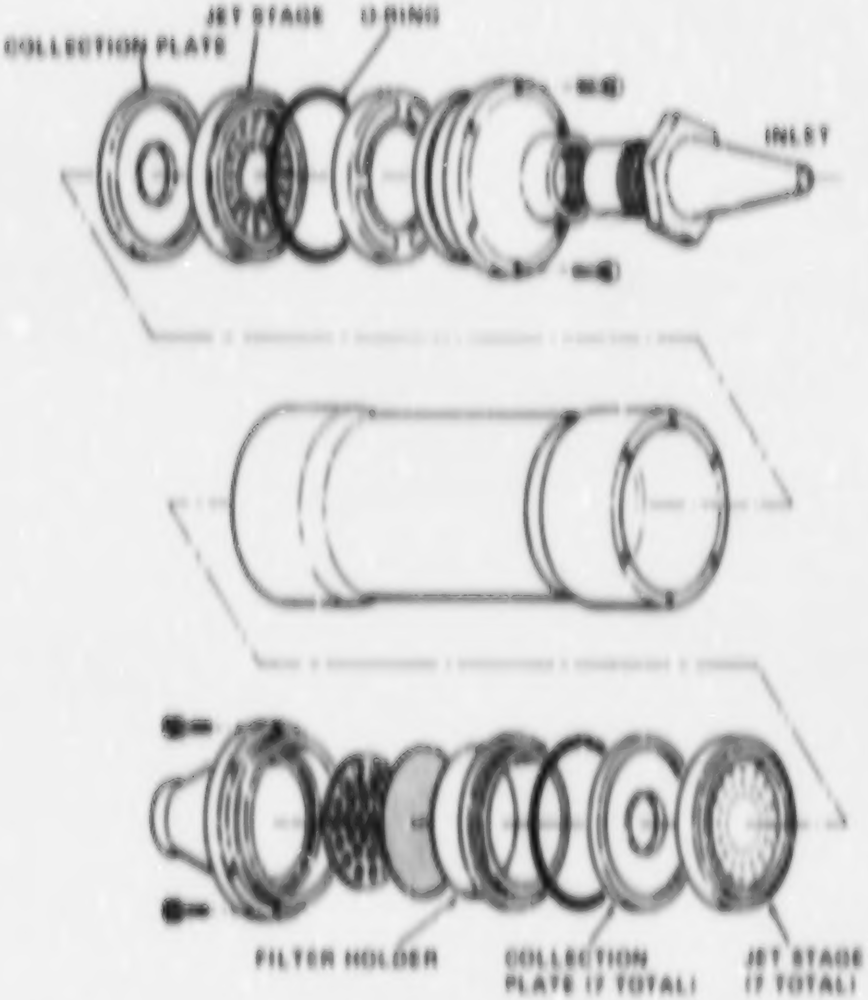

JET STAGE
O RING
COLLECTION PLATE
INLET
FILTER HOLDER
COLLECTION PLATE (7 TOTAL)
JET STAGE (7 TOTAL)

*Figure 23. University of Washington Mark III Source Test Cascade Impactor.*

## TABLE I

## COMMERCIAL CASCADE IMPACTOR SAMPLING SYSTEMS

| Name | Nominal Flow rate (cm$^3$/sec) | Substrates | Manufacturer |
|---|---|---|---|
| Andersen Stack Sampler (Precollection Cyclone Avail.) | 236 | Glass Fiber (Available from manufacturer) | Andersen 2000, Inc.<br>P.O. Box 20769<br>Atlanta, GA 30320 |
| Univ. of Washington Mark III Source Test Cascade Impactor (Precollection Cyclone Avail.) | 236 | Stainless Steel Inserts, Glass Fiber, Grease | Pollution Control System Corp.<br>321 Evergreen Bldg.<br>Renton, WA 98055 |
| Brink Cascade Impactor (Precollection Cyclone Avail.) | 14.2 | Glass Fiber, Aluminum, Grease | Monsanto EnviroChem Systems, Inc.<br>St. Louis, MO 63166 |
| Sierra Source Cascade Impactor - Model 226 (Precollection Cyclone Avail.) | 118 | Glass Fiber (Available from manufacturer) | Sierra Instruments, Inc.<br>P.O. Box 909<br>Village Square<br>Carmel Valley, CA 93924 |
| MRI Inertial Cascade Impactor | 236 | Stainless Steel, Aluminum, Mylar, Teflon. Optional: Gold, Silver, Nickel | Meteorology Research, Inc.<br>Box 637<br>Altadena, CA 91001 |

the inlet and a high flow rate impactor at the outlet. The impactors are then operated at their respective optimum flow rates, and the sampling times are dictated by the time required to collect weighable samples on each stage without overloading any single stage.

*Series cyclone particulate sampling techniques* - Prototype series cyclone sampling systems have been developed for industrial source sampling.[7] In general, series cyclones are easy to use, trouble-free, and efficient collectors of large quantities of size segregated particulate. Their main drawbacks are that their size limits the number of size segregated samples during each test that can be obtained as compared to most commercial impactors and an accurate theory of operation has not been developed. However, the ability to collect large quantities of sized material for analysis makes these devices irreplaceable for some applications.

The Source Assessment Sampling System (SASS) incorporates three cyclones and a back up filter.[19] A schematic of this system is illustrated in Figure 24. It is operated at a flow rate of 3065 $cm^3$/sec (6.5 $ft^3$/min) with approximate cyclone cut points of 10, 3, and 1 micrometer aerodynamic diameter and a gas temperature of 205°C. Besides obtaining information on the particle size distribution, this system collects gram quantities of particles for later chemical or biological analyses. The SASS train is large and requires extractive sampling through a heated probe. The cyclones are mounted in an oven to keep the air stream at stack temperature or above the dew point until the particles are collected. This system is supplied with nozzles, a probe-pitot-tube-thermocouple assembly, cyclones, back up filter, oven, a gas conditioning chamber, and a flow metering device pump adapted from the Aerotherm High Volume Sampling System.

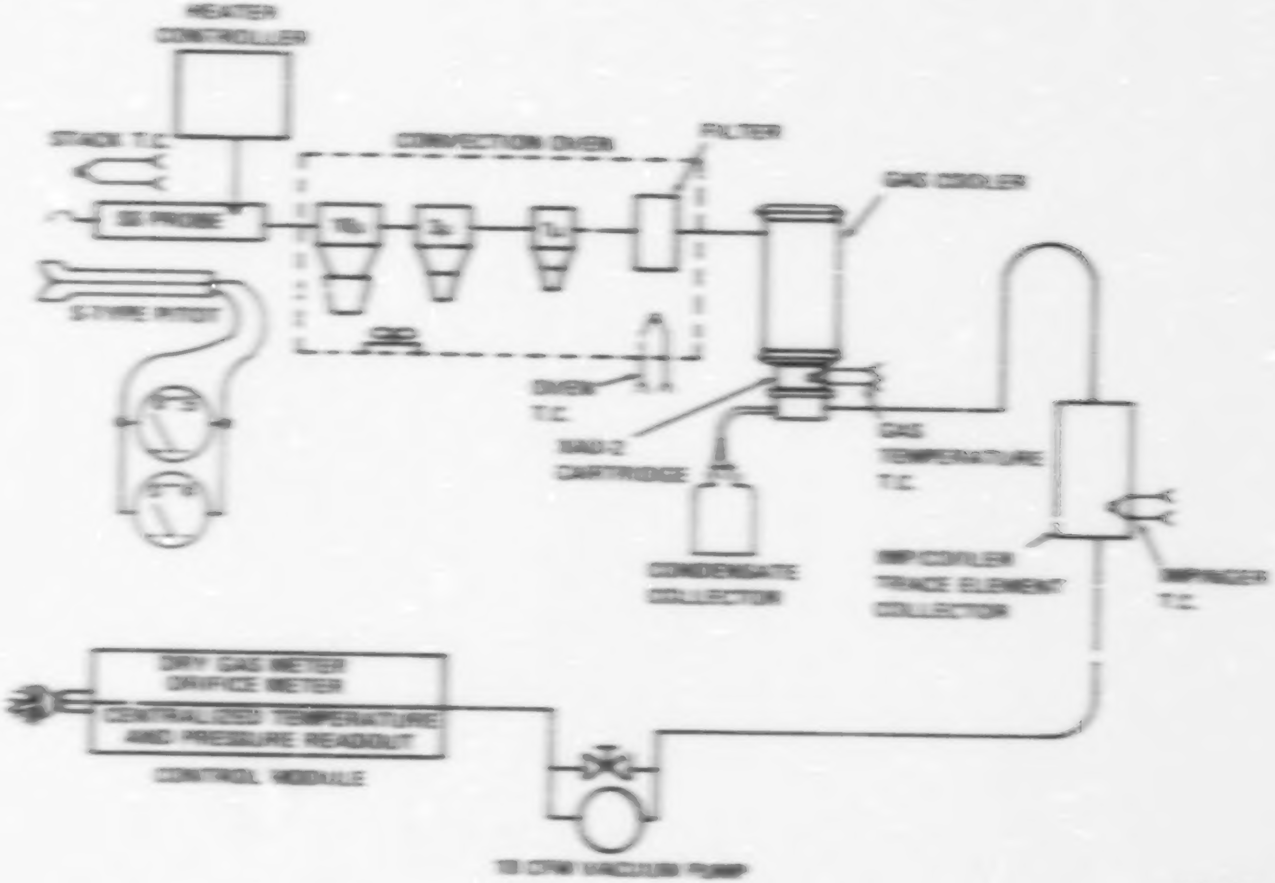

HEATER CONTROLLER
STACK T.C.
SS PROBE
S-TYPE PITOT
CONVECTION OVEN
10μ
3μ
1μ
FILTER
GAS COOLER
OVEN T.C.
XAD-2 CARTRIDGE
GAS TEMPERATURE T.C.
CONDENSATE COLLECTOR
IMP/COOLER TRACE ELEMENT COLLECTOR
IMPINGER T.C.
DRY GAS METER ORIFICE METER
CENTRALIZED TEMPERATURE AND PRESSURE READOUT
CONTROL MODULE
10 CFM VACUUM PUMP

Figure 24. Schematic of the Source Assessment Sampling System.

Cyclone calibration details are furnished along with equations for calculating approximate cyclone cutpoints for operating conditions other than those measured during the calibration procedure.

It is mandatory that the gas velocity and temperature through the cyclones be maintained at a constant setting while sampling, because the cyclone cut points are dependent upon the gas flow rate and temperature. This usually means that periods of nonisokinetic sampling may occur. Depending on the magnitude of fluctuations in the velocity of the sampled stream, this may or may not introduce significant errors in the sizing process.

Southern Research Institute, under EPA sponsorship, has designed and built a prototype three-stage series cyclone for in-stack use. A sketch of this system is shown in Figure 25. It is designed to operate at 472 $cm^3/sec$ (1 $ft^3/min.$). The calibrated cut points for these cyclones are 3.0, 1.6, and 0.6 micrometer aerodynamic at 21°C. A 47 mm Gelman filter holder is used as a back up filter after the last cyclone. This series cyclone system was designed for in-stack use and requires a six inch sampling port. A sampling system similar to that for a high flow rate impactor is usually adequate, although a more powerful pump may be required under some sampling situations. As with the SASS train, a constant flow rate through the cyclone system is required to maintain stable cyclone cut points.

Figure 26 shows a second generation EPA/Southern Research series cyclone system under development which contains five cyclones and a back up filter. It is a compact system and will fit through 4 inch diameter ports. The

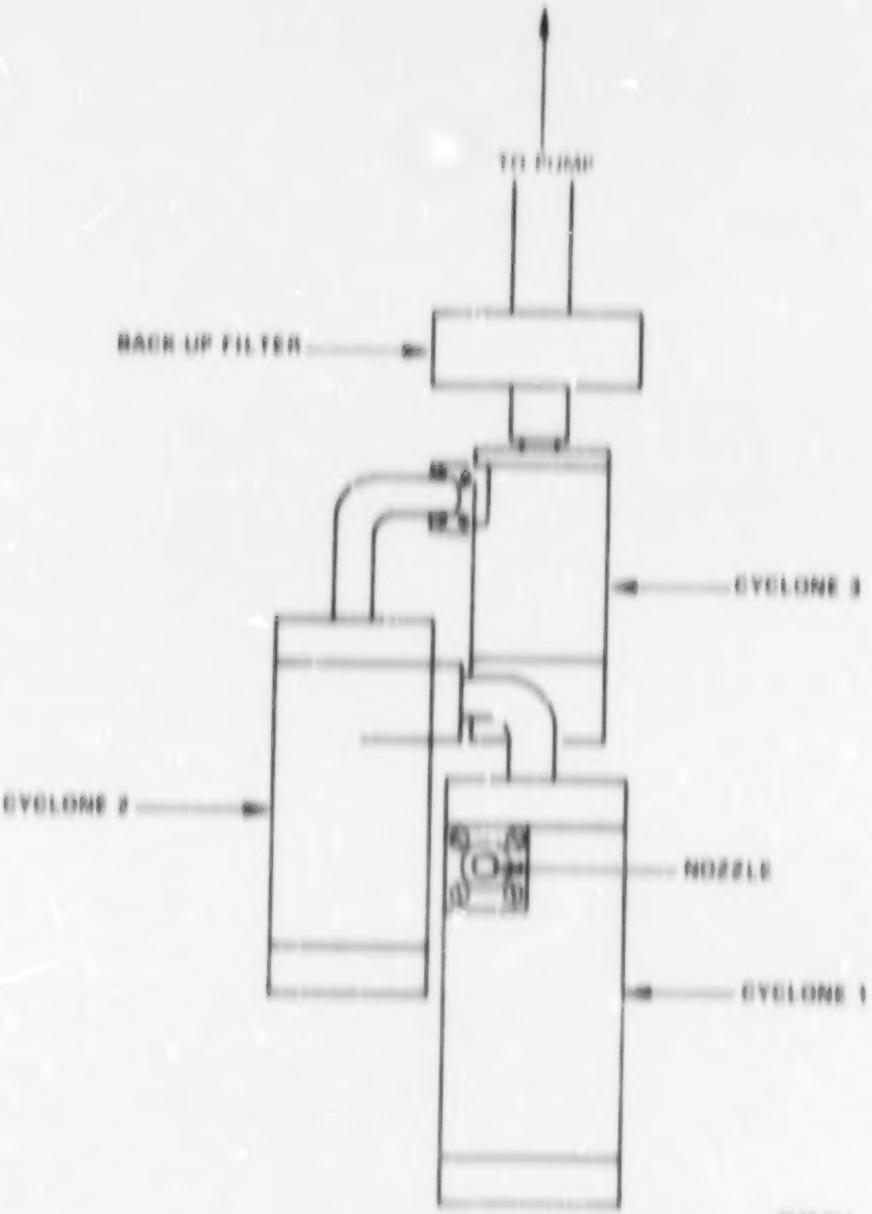

TO PUMP
BACK UP FILTER
CYCLONE 3
CYCLONE 2
NOZZLE
CYCLONE 1

*Figure 25. Three Stage Series Cyclone System.*

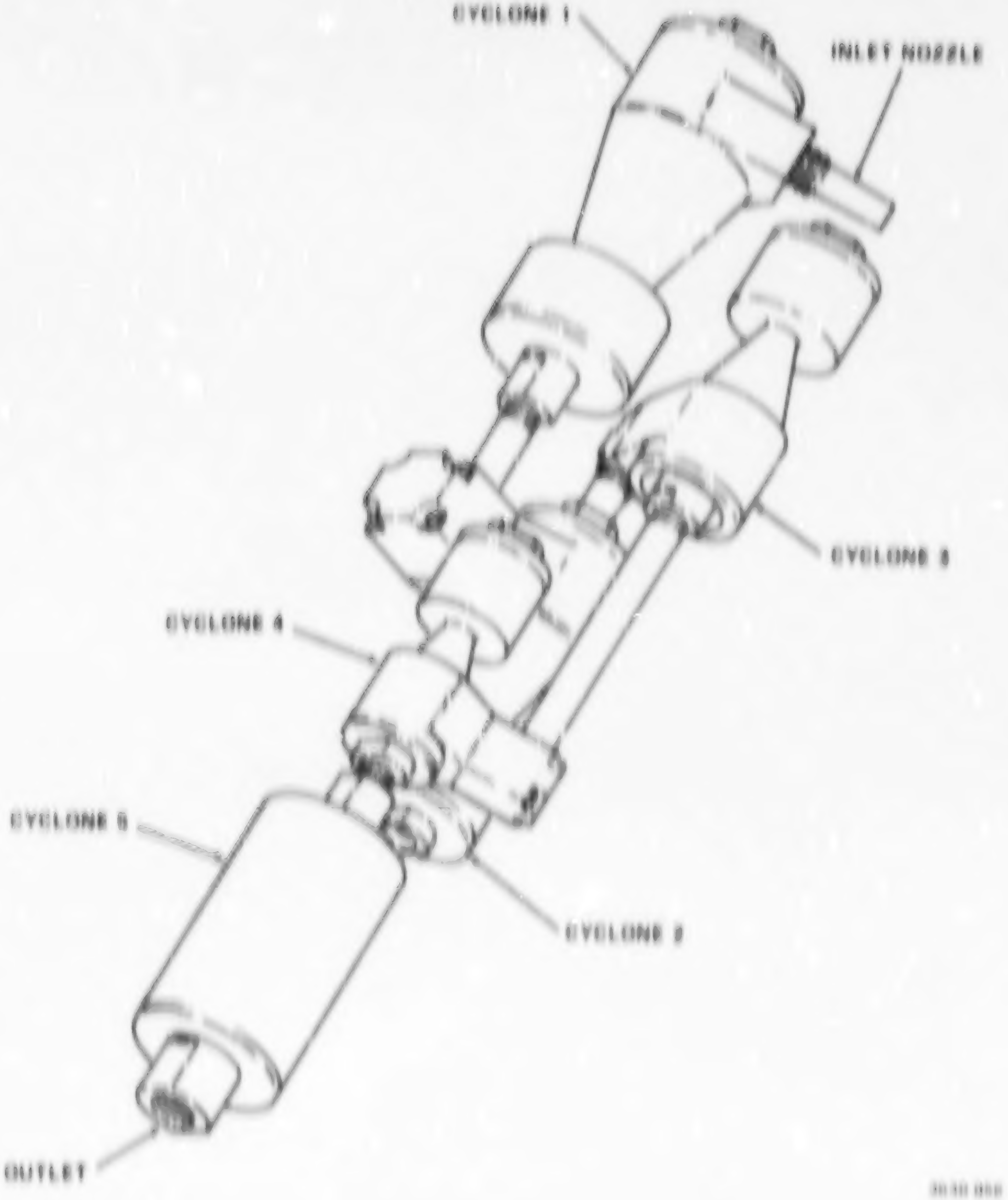

CYCLONE 1
INLET NOZZLE
CYCLONE 3
CYCLONE 4
CYCLONE 5
CYCLONE 2
OUTLET

*Figure 26. Five Stage Series Cyclone System.*

initial prototype was made of anodized aluminum with stainless steel connecting hardware. A second prototype, for in-stack evaluation, is made of titanium.

Figure 27 shows laboratory calibrations of the five cyclone prototype system. The cut points, at the test conditions are 0.32, 0.6, 1.1, 2.6, and 7.5 µm. A continuing research program includes studies to investigate the dependence of the cyclone cut points upon the sample flow rate and temperature so that the behavior of the cyclones at stack conditions can be predicted more accurately.

Small cyclone systems appear to be practical alternatives to cascade impactors as instruments for measuring particle size distributions in process streams. Cyclones offer several advantages:

Large, size segregated samples are obtained.

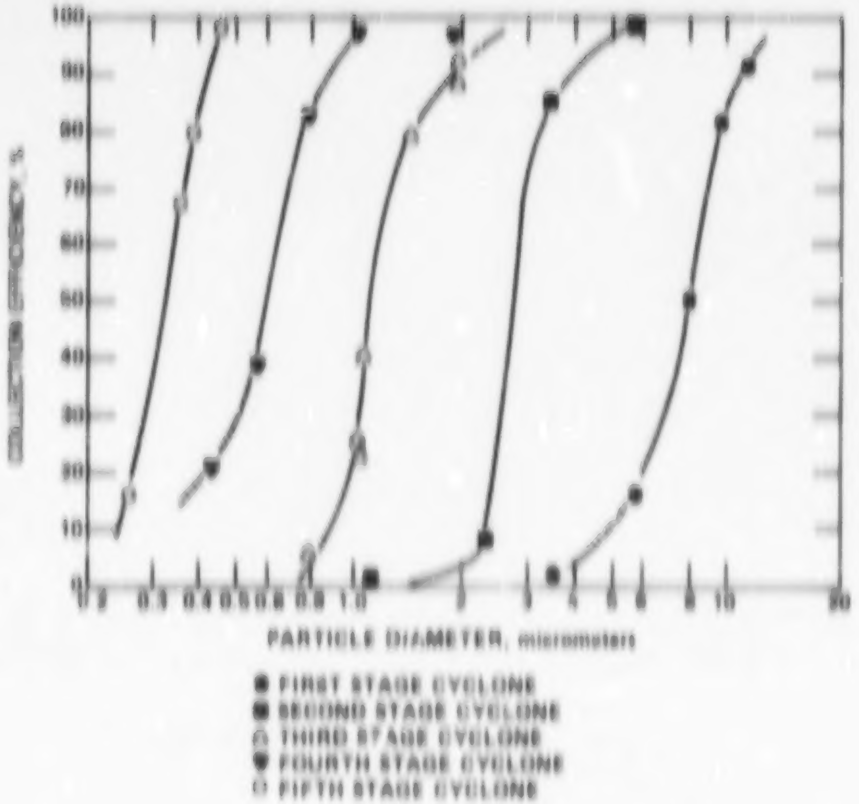
There are no substrates to interfere with analyses.

They are convenient and reliable to operate.

They allow long sampling times under high mass loading conditions for a better process emission average.

They may be operated at a wide range of flow rates without particle bounce or reentrainment.

On the other hand, there are some negative aspects of cyclone systems which require further investigation:



1630 017

*Figure 27. Laboratory Calibration for the Five Stage Series Cyclone System. (472 cm³/sec, particle density=1.0 gm/cm³)*

Unduly long sampling times may be required to obtain large samples at relatively clean sources.

The existing theories do not accurately predict cyclone performance.

Cyclone systems are bulkier than impactors and may require larger ports for in-stack use.

As discussed above, cyclones are now used on an experimental basis by the EPA and EPA contractors. If current research programs are successful in developing a better understanding of cyclone behavior, they may play a very important role in control device evaluation.

### 2.3.3 Optical Measurement Techniques

The basic operating principle for one type of optical particle counter is illustrated in Figure 28. Light is scattered by individual particles as they pass through a small viewing volume, the intensity of the scattered light being measured by a photodetector. The sizes of the particles determine the amplitude of the scattered light pulses, and the rate at which the pulses occur is related to the particle concentration. Thus, a counter of this type gives both size and concentration information. The simultaneous presence of more than one particle in the viewing volume is interpreted by the counter as a larger single particle. To avoid errors arising from this effect, dilution to about 300 particles/$cm^3$ is generally necessary. Errors in counting rate also occur as a result of electronics deadtime and from statistical effects resulting from the presence of high concentrations of sub-countable ($D < 0.3$ µm) particles in the sample gas stream.[11] The intensity of the scattered light depends upon

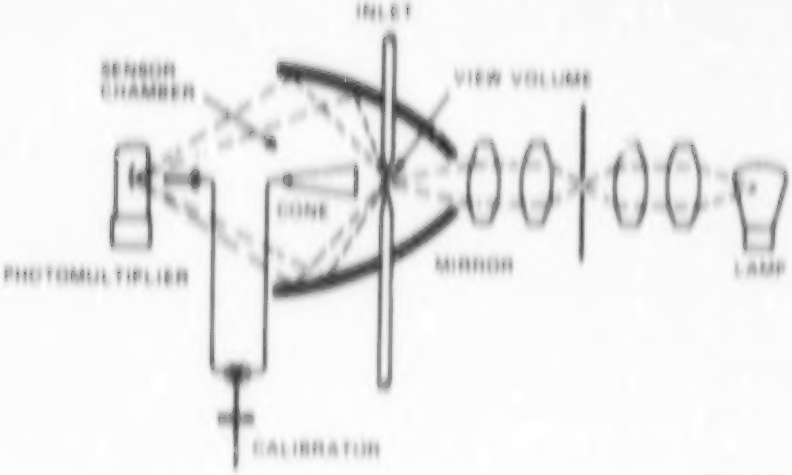

INLET
SENSOR CHAMBER
VIEW VOLUME
CONE
PHOTOMULTIPLIER
MIRROR
LAMP
CALIBRATOR

*Figure 28. Operating principle for an optical particle counter. Courtesy of Climet Instruments Company.*

the viewing angle, particle index of refraction, particle optical absorptivity, and shape, in addition to the particle size. The schematic in Figure 28 shows a system which utilizes "integrated near forward" scattering. Different viewing angles might be chosen to optimize some aspect of the counter performance. For example, near forward scattering minimizes the affect of variations in the indicated particle size with index of refraction, but for this geometry, there is a severe loss of resolution for particle diameters near 1 μm. Right angle, or 90° scattering smooths out the response curve, but the intensity is more dependent on the particle index of refraction.

Available geometries are:

| | |
|---|---|
| Bausch & Lomb 40-1 | Near Forward Scattering |
| Royco 220 | Right Angle |
| Royco 245, 225 | Near Forward |
| Climet CI-201, 208 | Integrated Near Forward |

Optical particle counters have not been used extensively in stack sampling because they cannot be applied directly to the effluent gas stream. The sample must be extracted, cooled, and diluted; a procedure which requires great care to avoid introducing serious errors into calculations of the particle size distribution. The main advantage of optical counters is the capability of observing emission fluctuations in real time. After extraction, the useful particle size range is approximately 0.1 to 1.5 μm.

### 2.3.4 Ultrafine Particle Sizing Techniques

There are two physical properties of ultrafine particles (diameter < 0.5 μm) which are size dependent and which can be predicted with sufficient accuracy under controlled conditions to be used to measure particle size. These are the particle diffusivity and electrical mobility. Although ultrafine particle size distribution measurements are still in a developmental stage, instruments are available which can be used for this purpose, and some field measurements have been made. A practical limitation on the lower size limit for this type of measurement is the loss of particles by diffusion in the sampling lines and instrumentation. These losses are excessive for particle sizes below about 0.01 μm where the samples are extracted from a duct and diluted to concentrations within the capability of the sensing devices.

Diffusional sizing - Diffusion batteries may consist of a number of long, narrow, parallel channels, a cluster of small bore tubes, or a series of screens. Figure 29a shows a typical parallel channel diffusion battery, and Figure 29b shows the aerosol penetration characteristics of this geometry at two flow rates. The parallel plate geometry is convenient because of ease of fabrication and the availability of suitable materials, and also because sedimentation can be ignored if the slots are vertical, while additional information can be gained through settling, if the slots are horizontal.

Sinclair[16] and Breslin et al[17] report success with more compact, tube-type and screen-type arrangements in laboratory studies and a commercial version of Sinclair's geometry is available.* Although the screen-type diffusion battery must be calibrated empirically, it offers convenience in cleaning

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*Thermo-Systems, Inc., 2500 N. Cleveland Ave., St. Paul, Minnesota 55113.

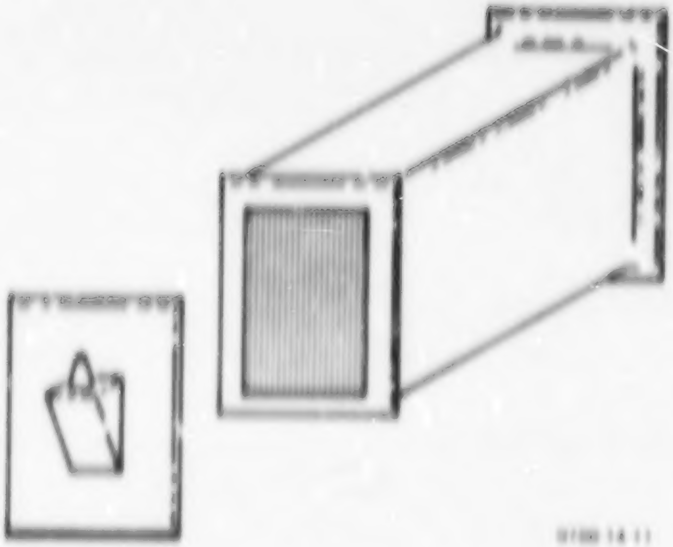

*Figure 29a. Parallel plate diffusion battery.*

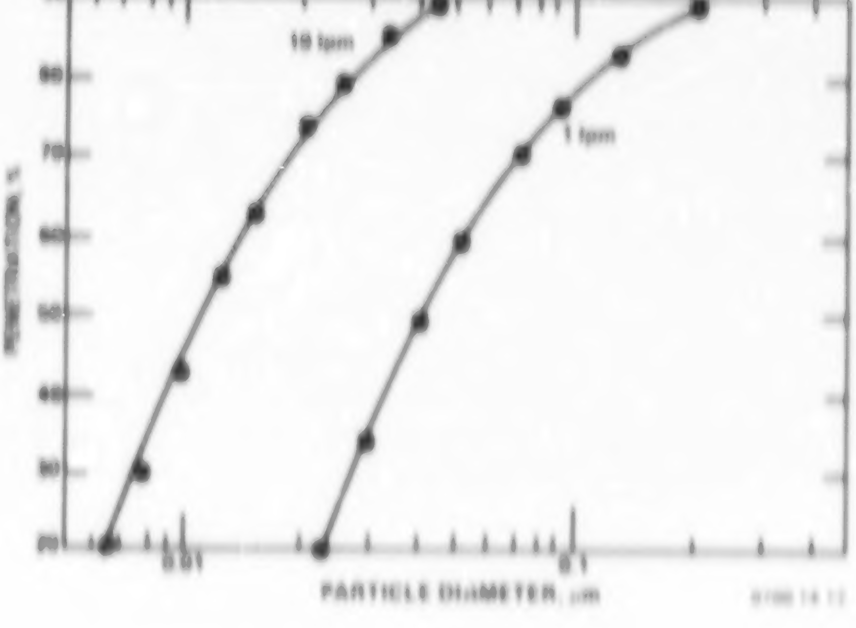

*Figure 29b. Parallel plate diffusion battery penetration curves for monodisperse aerosols (12 channels, 0.1 × 10 × 48 cm).*

and operation, and compact size. Figure 30 shows Sinclair's geometry. This battery is 21 cm long, approximately 4 cm in diameter, and weighs 0.9 kg.

Variations in the length and number of channels (tubes, or screens) and in the aerosol flow rate are used as means of measuring the number of particles in a selected size range. As the aerosol moves in streamline flow through the channels, the particles diffuse to the walls at a predictable rate, depending on the particle size and the diffusion battery geometry. It is assumed that every particle which reaches the battery wall will adhere, therefore, only a fraction of the influent particles will appear at the effluent of a battery. It is only necessary to measure the total number concentration of particles with a condensation nuclei counter at the inlet and outlet to the diffusion battery under a number of conditions in order to calculate the particle size distribution.

Diffusional measurements are less dependent upon the aerosol parameters than the other techniques discussed and perhaps are on a more firm basis from a theoretical standpoint.

Disadvantages of the diffusional technique are the bulk of the diffusional batteries, although advanced technology may alleviate this problem; the long time required to measure a size distribution; and problems with sample conditioning when condensible vapors are present.

Electrical particle counters - The most complete set of experiments performed in order to determine the relationship between particle size and charge were reported by Hewitt[17]

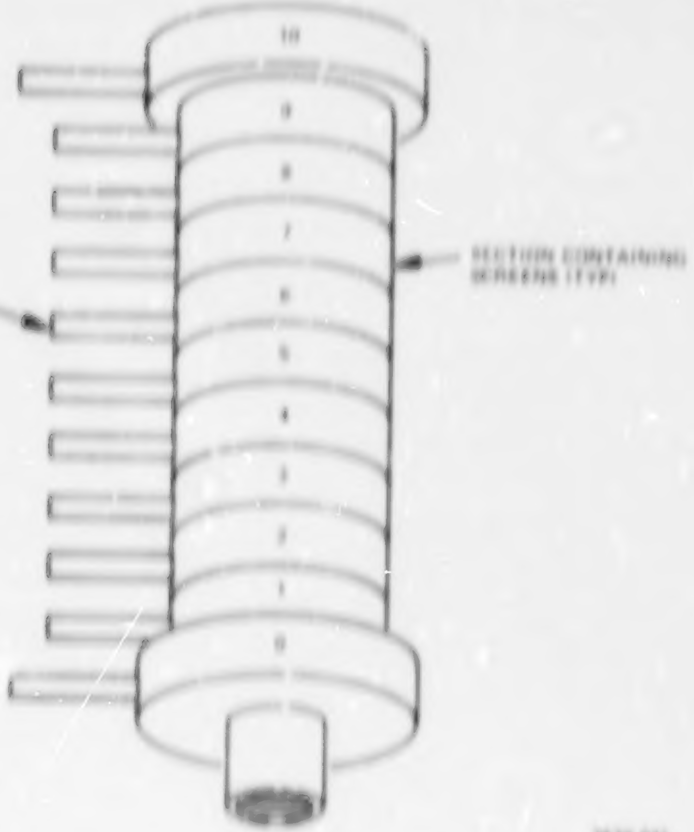
10
9
8
7
6
5
4
3
2
1
0
SAMPLING PORT (TYP)
SECTION CONTAINING SCREENS (TYP)

*Figure 30.* *Screen type diffusion battery. The battery is 21 cm long, 4 cm in diameter, and contains 55, 635 mesh stainless steel screens.*

in 1957. These experiments confirmed theoretical predictions that there exists a unique charging rate for each particle size if the charging region is homogeneous with respect to space charge density and electric field.

In the course of his work, Hewitt developed a mobility analyzer, shown in Figure 31. Charged particles enter through the narrow annular passage A, and experience a radial force toward the central cylinder due to the applied field. By moving the sampling groove B, axially, or by varying the magnitude of the applied field, the mobility of the charged particles can be measured. In Hewitt's experimental work, the particle size was known, and the mobility determined the charge. If, however, the particle charge were known, the mobility would determine the particle size. This concept has been used by Whitby, Liu, *et al*[12] at the University of Minnesota to develop a series of Electrical Aerosol Analyzers (EAA). A ruggedized field test unit based on the earlier University of Minnesota designs is now commercially available.* A schematic of this system is shown in Figure 32.

The EAA has the distinct advantage of very rapid data acquisition compared to diffusion batteries and condensation nuclei counters (two minutes as opposed to two hours for a single size distribution analysis.)

Disadvantages of this type of measurement system are difficulties in predicting the particle charge, and the fraction of the particles bearing a charge, with sufficient accuracy, and the requirement for sample dilution when making particle size distribution measurements in flue gases.

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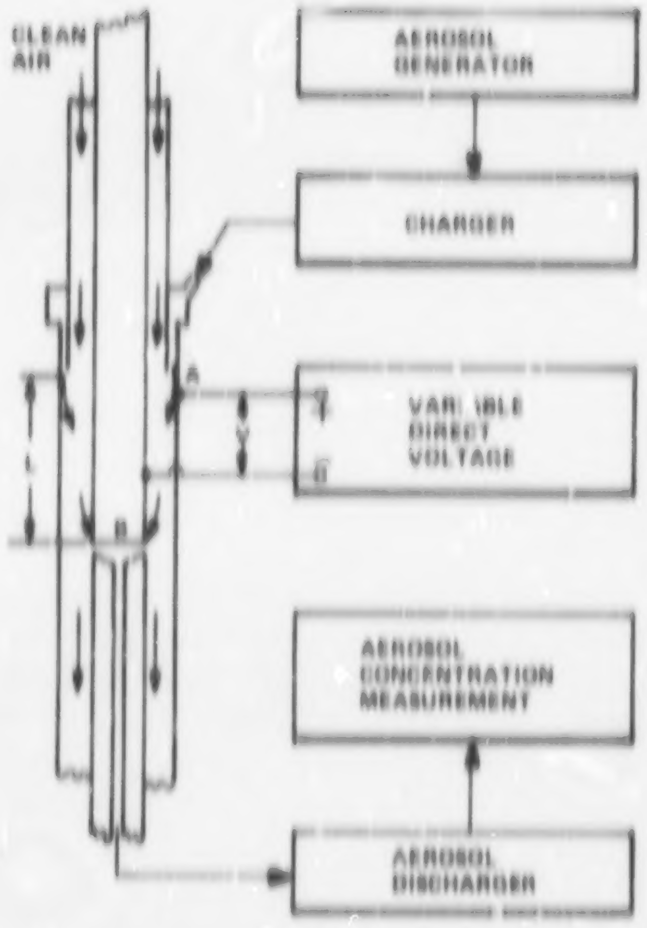
*Thermo-Systems, Inc., 2500 N. Cleveland Ave., St. Paul, Minnesota 55113.


CLEAN AIR
AEROSOL GENERATOR
CHARGER
A
L
V
B
VARIABLE DIRECT VOLTAGE
AEROSOL CONCENTRATION MEASUREMENT
AEROSOL DISCHARGER

0700 14 13

*Figure 31.* *Coaxial cylinder mobility analyser. Charged aerosol enters at A. Sampling groove is at B in inner cylinder, which is adjusted axially. After Hewitt (14).*

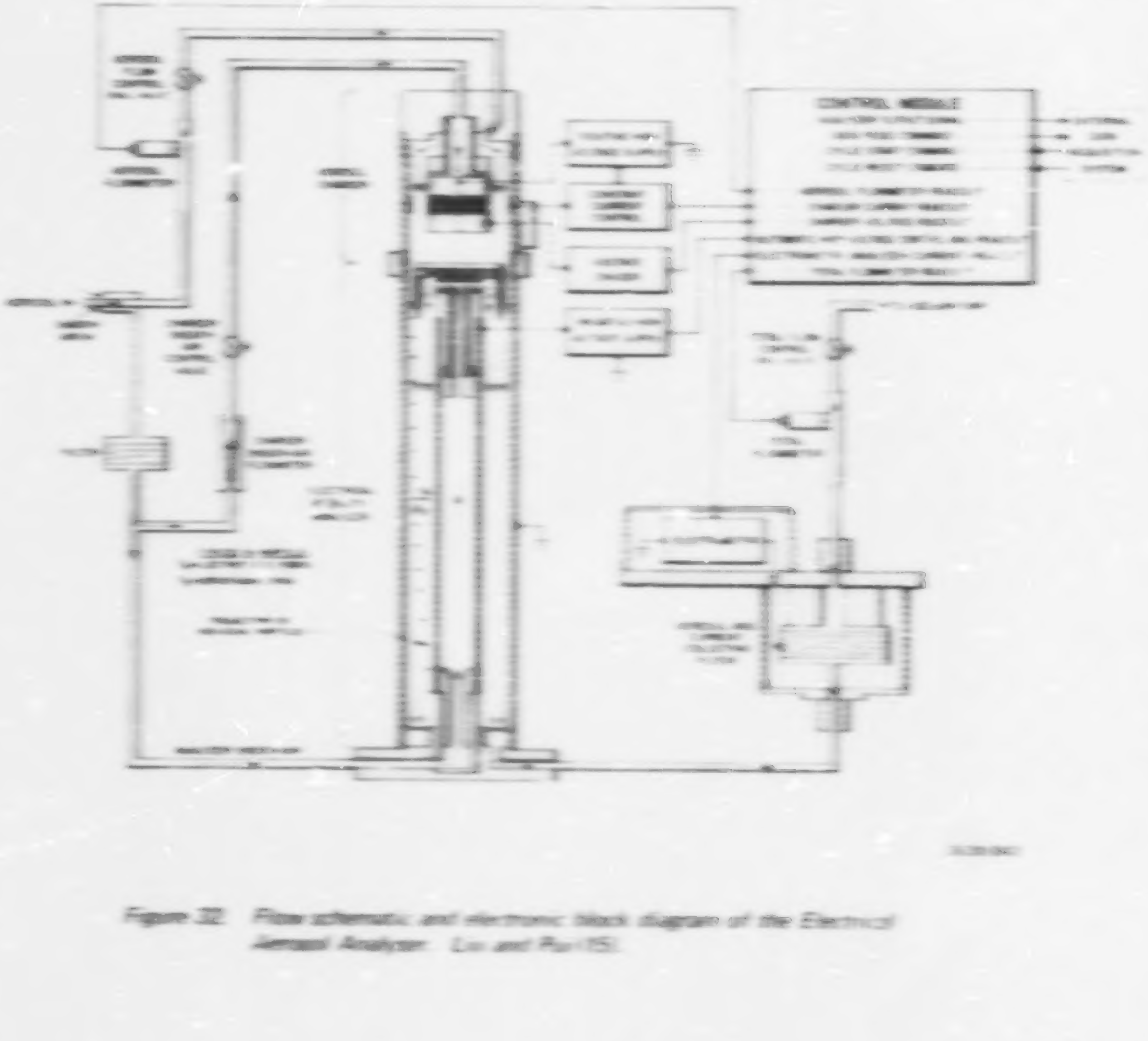

CONTROL MODULE

Figure 32. Flow schematic and electronic block diagram of the Electrical Aerosol Analyzer (Liu and Pui (15).

Figures 33, 34, and 35 show typical particle size distributions and a precipitator fractional efficiency curve measured in the field with impactors, an optical particle counter, and a diffusional ultrafine apparatus.

The details of sampling with optical particle counters, diffusion batteries with condensation nuclei counters, and EAA using extractive sampling and dilution techniques are presented in Appendix D.

## 2.4 PARTICULATE RESISTIVITY MEASUREMENTS

### 2.4.1 General Discussion

The resistivity of the dust collected in a precipitator has a direct influence on the efficiency of collection. If the resistivity is greater than about $5 \times 10^{11}$ ohm-cm, the electrical field developed in the collected particulate layer can exceed the breakdown field strength. Excessive spark rates and back corona can occur, necessitating precipitator operation at lower current densities with resultant degraded performance. If the particulate resistivity is less than about $10^{7}$ ohm-cm, the electrical forces holding the dust to the collection plates will be low. Excessive reentrainment can occur yielding lower performance. Therefore, to perform a complete evaluation of a precipitator installation, it is important to determine the particulate resistivity.

The particulate resistivity can be determined in the laboratory or in the field. In the laboratory, the dust sample is placed in a suitable test cell, and the current passed through the dust layer under a given voltage is

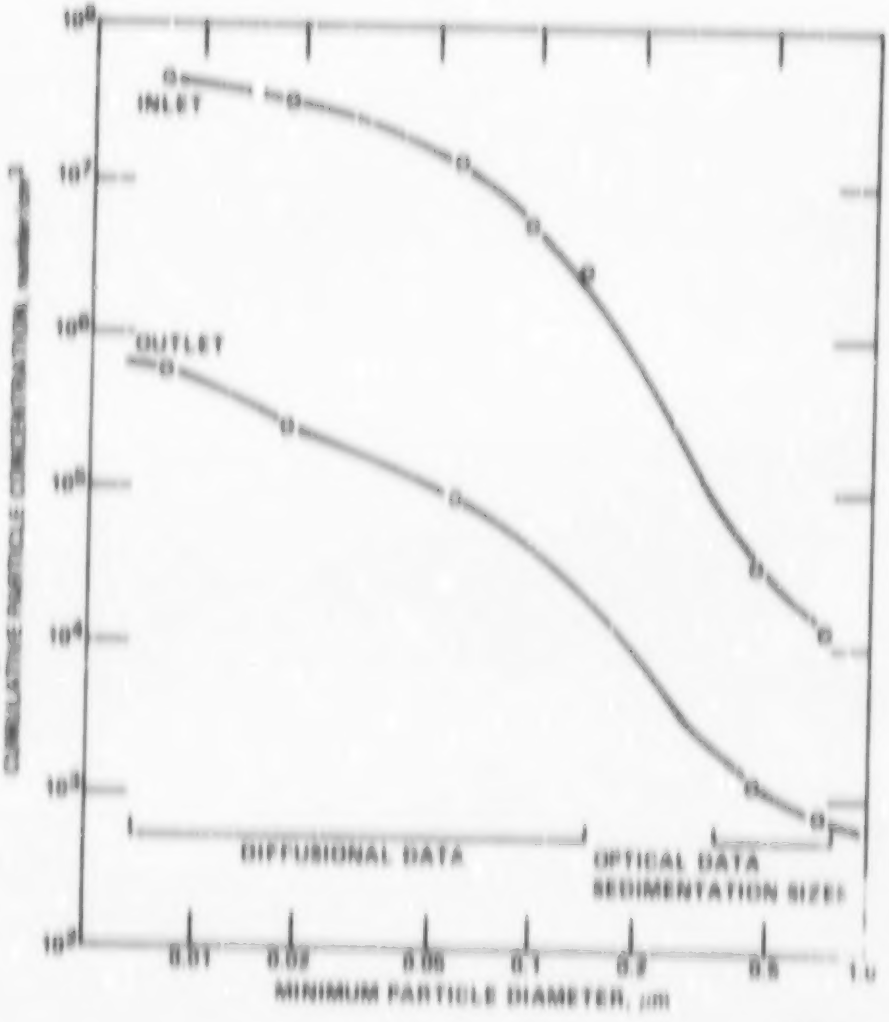


*Figure 33. Cumulative particle number concentration. Diffusional and optical data are indicated.*

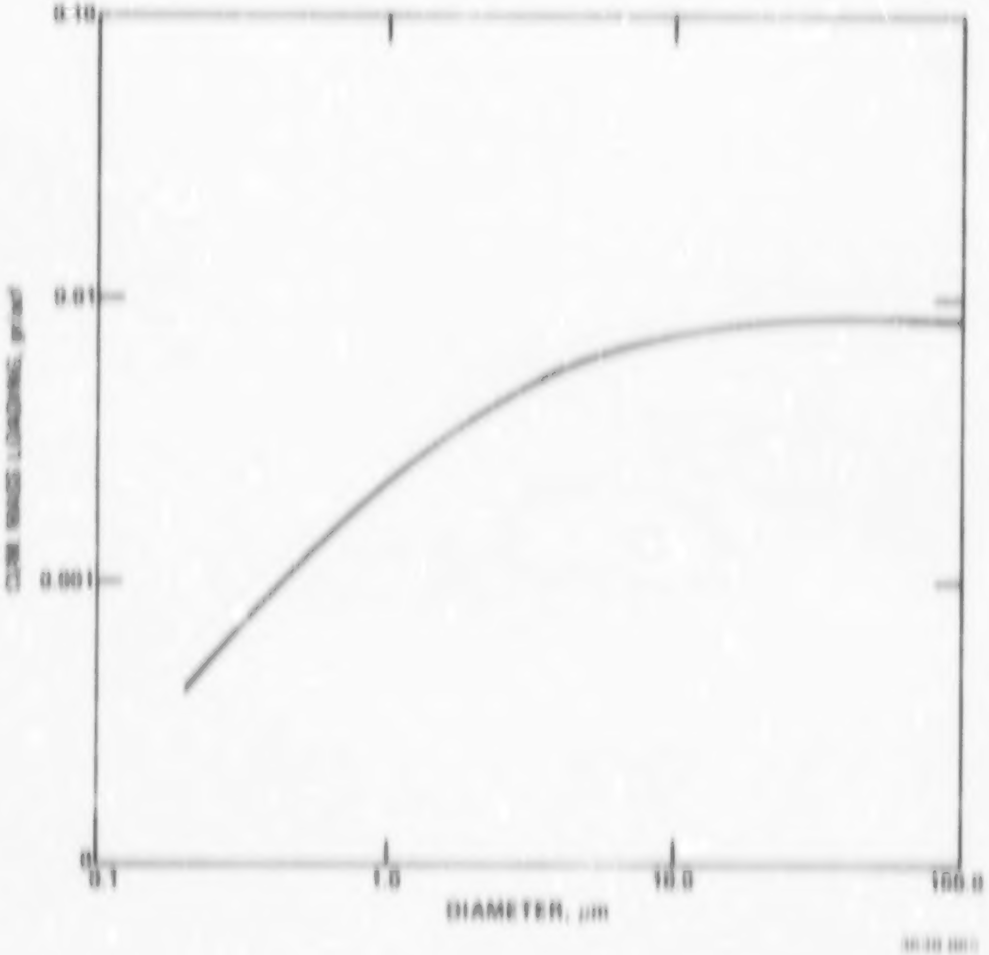


*Figure 34. Precipitator outlet particle size distribution obtained with an Andersen Mark III Stack Sampler.*

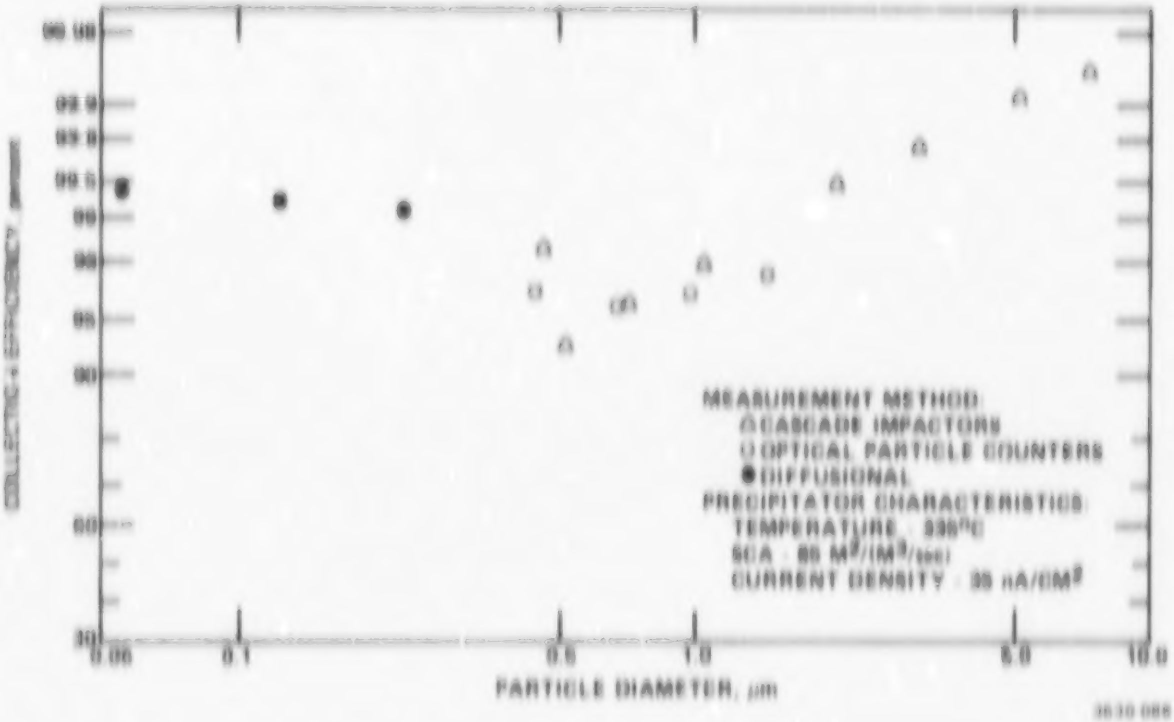


*Figure 36. Measured fractional efficiencies for a hot side electrostatic precipitator installed on a pulverized coal boiler. The operating parameters are indicated.*

measured. Resistivity is calculated from these values and known geometrical factors. For field determinations of resistivity, a variety of resistivity probes are available. These probes are inserted into the gas stream to extract or collect the dust either electrostatically or inertially. Resistivity can be determined from the V-I curves generated if electrostatic collection is used or from the measured values of current passing through the collected dust layer under given applied voltages and geometrical factors. Since the resistivity is determined while the dust is in its original or natural environment, the data are referred to as _in situ_ resistivity values. The equipment and procedures used for measuring resistivity in the laboratory and field have been recently reviewed.[14]

The question of whether laboratory or _in situ_ resistivity data should be acquired depends upon many factors. The question is somewhat simplified when the viewpoint is restricted to the evaluation of existing precipitator performance characteristics.

First, consider a "hot side" installation operating in the temperature range of 200°C to 400°C. It is generally accepted that in this temperature range the resistivity is not significantly influenced by environmental factors. Therefore it is assumed that _in situ_ and laboratory measurements will give comparable results. However, too little _in situ_ information is available to substantiate this assumption. Since high temperature probes are not presently generally available, laboratory data are usually used.

If a "cold side" installation operating between 100°C and 200°C is to be evaluated, the _in situ_ data are recommended. In this temperature range the resistivity can be affected greatly by the environment. Furthermore in evaluating precipitator performance, one generally desires data taken simultaneously with the measurement of other operating characteristics. Laboratory data have been successfully used for situations in which no conditioning agents were being used and the particulate was generated from low sulfur coals. There is an ongoing attempt by several investigators to develop a sophisticated laboratory technique which will allow one to utilize laboratory resistivity data when evaluating "cold side" precipitator operation.

### 2.4.2 Laboratory Determination of Particulate Resistivity

There are two almost identical standards available for the determination of bulk particulate resistivity in the laboratory. These are the ASME Power Test Code 28[17] and the APCA Informative Report No. 2.[18] The preferable dust specimen for these tests is an isokinetically obtained high volume sample taken at the precipitator inlet. Samples used include: hopper samples proportionately blended based on a known precipitator efficiency and the number of fields, samples from individual hoppers, and samples from ash storage facilities. The hardware, environmental control, and procedures recommended by the above standards are given in detail in Appendix E. Figure 36 shows typical resistivity data as a function of temperature and water concentration in an air environment obtained using these standards with exception of recommended voltage gradient. Many laboratories use equipment and procedures significantly different from the above standards. These variations have developed with

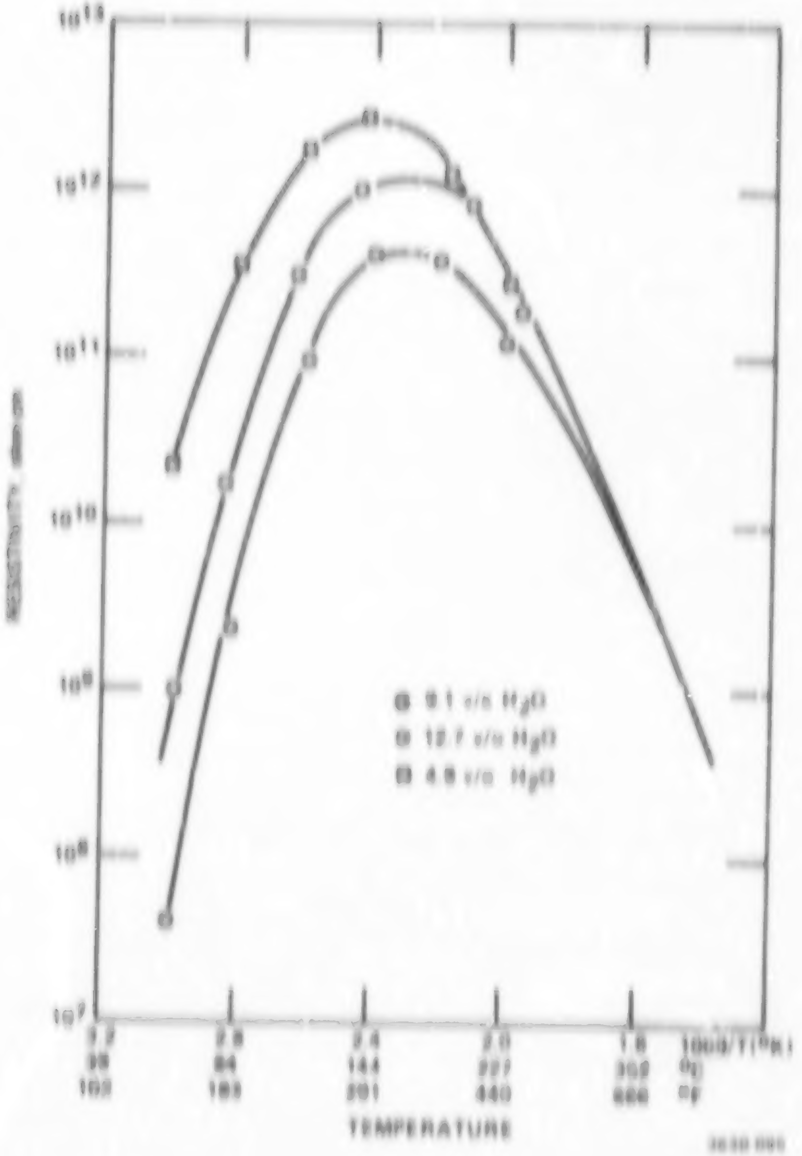


*Figure 36. Typical resistivity versus temperature curves for fly ash at three water vapor concentrations.*

respect to specific research goals and in efforts to eliminate observed shortcomings. An example of an alternate procedure is also detailed in Appendix E.

### 2.4.3 In Situ Particulate Resistivity Measurement

An *in situ* determination of particulate resistivity makes use of a point-to-plane probe which can be inserted directly into the gas stream upstream of the precipitator. A high voltage is applied to the point-to-plane electrode system such that a corona is formed in the vicinity of the point. The dust particles are charged by ions created in the corona, and precipitated by electrical forces onto the collection plate. Thus, the probe is intended to simulate the behavior of a full-scale electrostatic precipitator and to measure the resistivity of the dust in a way that simulates the operating condition.

In the point-to-plane technique, two methods of making resistivity measurements on the same sample may be used. The first is the "V-I" method. In this method, voltage-current curves for the point-plane system are obtained before and after the electrostatic deposition of the dust.

Alternatively, a disc the same size as the collecting disc is lowered onto the collected sample. Increasing voltages are then applied to the dust layer and the currents recorded until the dust layer breaks down electrically and sparkover occurs. More information can be found in the paper by Nichols[16] concerning the advantage and disadvantage to using *in situ* resistivity probes.

Several types of resistivity probes have been developed and are currently available. In Appendix F, full details for the use of one particular probe are given.

## 2.5 PROCESS EFFLUENT GAS ANALYSIS

### 2.5.1 General Discussion

In evaluating the performance of a precipitator it is advantageous to know something of the constituents of the effluent gas stream, excluding the particulate matter.

A gas analysis generally concerns the amount of $N_2$, $O_2$, CO, $CO_2$, and $H_2O$ in the effluent. These can usually be determined using an ORSAT apparatus or one of the EPA Stationary Source Test Methods developed for gas analysis. Experimental studies on modifying combustion and conditioning the particles to modify their resistivity, and the effect this has on emissions, has resulted in the need for measuring the amounts of several other gas constituents such as $NH_3$, $SO_2$, $SO_3$, $NO_x$, etc. These measurement methods are briefly described in the following section.

### 2.5.2 Qualitative Gas Analysis

Interviewing key plant personnel or obtaining the proper records should be sufficient to determine the qualitative nature of the process gas stream. This data should include the average fractional amounts of $CO_2$, CO, $O_2$, $N_2$, and $H_2O$ in the process effluent gas stream during normal operation. Depending on the industrial process under consideration, there may also be measurable amounts of $NH_3$, $SO_2$, $SO_3$, $NO_x$, HF, sulfuric acid mist, or other volatile substances present.

Other qualitative information which should be gathered includes the average gas temperature at the precipitator inlet and outlet, and the average actual and standard volumetric flow rates through the precipitator.

### 2.5.3 Quantitative Gas Analysis

Flue gas constituents normally specified for analysis are $N_2$, $O_2$, CO, and $CO_2$, and $H_2O$. In addition to these analysis, $SO_2$ and $SO_3$ concentrations are usually measured and sometimes the $NO_x$, HF or other vapor concentrations are determined.

Oxygen, CO, and $CO_2$ concentrations are measured with a commercial Orsat-type apparatus. Two Orsat-type analyzers are used to determine the oxygen content of the gas entering and leaving the precipitator simultaneously. Comparisons of the inlet and outlet oxygen concentration provides a check for leakage of gas into or out of the precipitator. Although in principle leakage can be determined from an examination of inlet and outlet gas velocity profiles, in practice, flow disturbances at available sampling locations often severely limit the accuracy with which flow determination can be made. Therefore, the simultaneous inlet and outlet oxygen determinations may be a more sensitive indicator of the physical integrity of the precipitator casing.

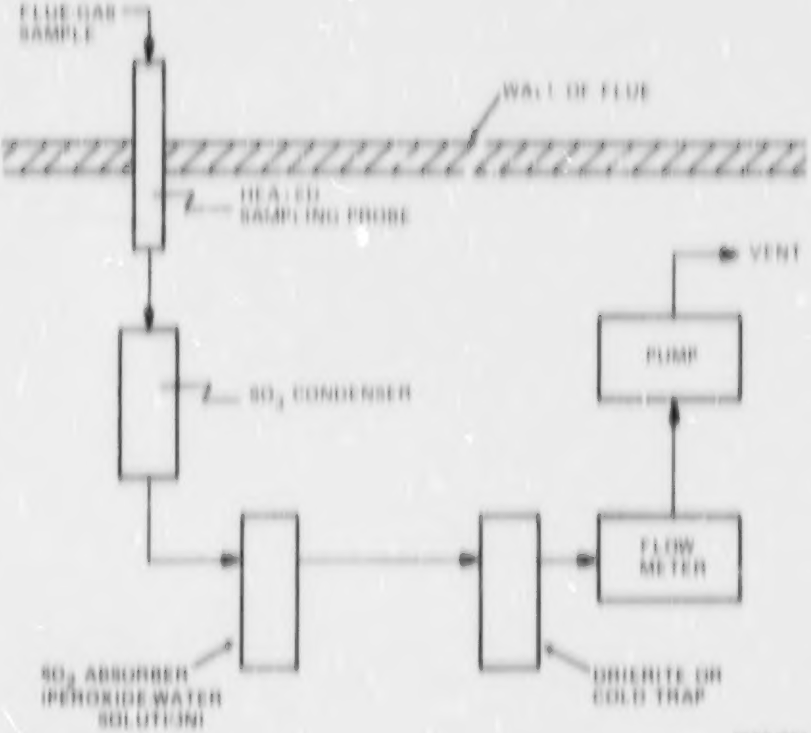
The Environmental Protection Agency has developed several Stationary Source Test Methods for the determination of various flue gas components. These methods, of course, are not the only way by which the quantity of the gases can be measured. There are many other acceptable analytical methods developed by different testing societies such as the ASME and ASTM. EPA Test Method Number 4 describes a procedure to determine the $H_2O$ content of the flue gas. EPA Test Method Number 6 can be used to determine the $SO_2$ content of the flue gas. EPA Test Method Number 7 explains a method for measuring the nitrogen oxide in the flue gas. The amount of sulfuric acid mist and $SO_2$ content can be determined using the EPA Test Method number 8.

Figure 37 illustrates a system not described in the Federal Register, which has been found to be accurate and convenient to use for measurements of $SO_2$ and $SO_3$ concentrations. This is the Controlled Condensation System developed for the EPA by TRW Systems Group[15] to accurately determine $SO_2$ and $SO_3$ concentrations in process gas streams. This procedure is applicable in high or low mass loading environments with temperatures up to 300°C and $SO_2$ concentrations up to 6000 ppm.

The Controlled Condensation System is based on the separation of $SO_3$ as $H_2SO_4$ from $SO_2$ by cooling the gas stream below the dewpoint of $H_2SO_4$, but above the $H_2O$ dewpoint. Cooling is accomplished by a water-jacketed coil where the $H_2SO_4$ is collected. Particulate matter is collected by a quartz filter mat inserted in the line prior to the condensation coil. The particulate filter system is maintained at a temperature of 288°C to insure that none of the $H_2SO_4$ will condense on the filter mat or filter holder.

The Controlled Condensation Coil (CCC) is a modified Graham Condenser. The water jacket is maintained at 60°C. This is adequate to reduce the flue gas below the dewpoint of the $H_2SO_4$. Following the CCC are two impingers for removing the $SO_2$ and $H_2O$. The $SO_2$ scrubber is a bubbler filled with a 3% solution of $H_2O_2$ in water. The water vapor is removed by a silica gel filled impinger. A vacuum pump with a capacity of 472 $cm^3$/sec is recommended. The total volume of gas sampled is measured with a dry gas meter.

The sampling rate/time is normally 135 $cm^3$/sec for one hour. An indication of the proper amount of sample comes



FLUE GAS SAMPLE
WALL OF FLUE
HEATED SAMPLING PROBE
VENT
PUMP
$SO_3$ CONDENSER
FLOW METER
$SO_2$ ABSORBER (PEROXIDE-WATER SOLUTION)
DRIERITE OR COLD TRAP

*Figure 37. Schematic diagram of apparatus for the collection of $SO_3$ by the condensation method.*

from watching the condenser coil. When the $H_2SO_4$ fog has crept about one-half to two-thirds of the way along the coil, the sampling can cease. After sampling has been completed, the probe and coil are rinsed with deionized $H_2O$ and the recovered solution is analysed in the lab. The amount of $H_2SO_4$ in the condensation coil and probe can be determined by a sulfate or $H^+$ titration. Because of its simplicity and sensitivity, the $H^+$ titration is preferred. This recommended acid/base titration uses Bromophenol Blue as the indicator, since the endpoint of the NaOH and $H_2SO_4$ titration falls near the pH range (3-4.6) of the Bromophenol Blue color change. Complete details can be found in the reference above.

# 3. DEVELOPMENT OF TEST PLANS FOR ELECTROSTATIC PRECIPITATOR EVALUATION

## 3.1 GENERAL DISCUSSION

Details of precipitator operation and particulate sampling procedures are given in the preceding sections and the appendices. This section is intended as a summary or outline to be used in the development of a test plan. Specific references to other more detailed sections are also included.

The scope of precipitator tests ranges from short visits to the site where the plant personnel are interviewed and the precipitator operating conditions are observed, to very elaborate experimental programs where it is attempted to completely characterize the properties of the flue gas aerosol and the electrical and mechanical conditions of the precipitator. Short visits may be sufficient to obtain data for diagnosis of precipitators which are malfunctioning, but more elaborate tests are almost always required if the precipitator is a poor or inadequate design, or if the properties of the dust limit performance.

For the purposes of developing test plans, it is possible to define three categories or levels of effort. These are denoted in this document by Level A, Level B, or Level C. A Level A test consists of the minimum effort that can be expected to yield positive results. It is the least expensive and also the most qualitative. Level C tests are comprehensive, expensive, and enough data is accumulated for definitive analyses. A Level B test falls between a Level

A and a Level B test in its expense and information gathered. A Level B evaluation includes all of the information obtained by a Level A evaluation and a Level C includes both Level A and Level B data. Table II is a flow diagram showing the major considerations for each level of effort.

## 3.2 LEVEL A PRECIPITATOR EVALUATION

Points to be considered in a Level A analysis are outlined in Table III. Information should be obtained on each of the items listed and references are shown on the table of the sections of this report which contain more detailed discussion.

Attempts should be made to determine if any of the power supplies show abnormally low readings. Any information that is available concerning the composition of the flue gas, the particle size distribution, and opacity should be obtained. *In situ* and laboratory measurements of the particulate resistivity are also included in a Level A test. Any evidence of excessive rapping losses should be noted.

## 3.3 LEVEL B PRECIPITATOR EVALUATION

The decisions involved in planning for a Level B precipitator evaluation are outlined in Table IV. A Level B evaluation includes all of the information obtained by a Level A evaluation plus measurements of the mass concentration at the precipitator inlet and outlet and quantitative gas analysis. Decisions must be made of the type of particulate sampling trains and gas sampling equipment to be used.

## TABLE II

## THREE LEVELS OF EFFORT FOR PRECIPITATOR EVALUATION

LEVEL

C B A

ENGINEERING AND OPERATING DATA

Precipitator Design & Operating Data
Qualitative Gas Composition
Particulate Resistivity (and approximate size distribution)
Opacity
Any Evidence of Rapping Reentrainment

Two men on site for two days. Two man weeks for analysis and reporting.

PARTICULATE MASS CONCENTRATION AND PRECIPITATOR EFFICIENCY

Pre-test Site Survey, Port Installation
Inlet and Outlet Mass Sampling
Quantitative Gas Analysis
Consider Test For Rapping Losses

Five men on site for 3 days. One man month for analysis and reporting.

PARTICLE SIZING AND PRECIPITATOR FRACTIONAL EFFICIENCY

Inlet and Outlet Particle Size Distribution

Ten to fifteen men on site for one to two weeks.
Six to twenty man months for analysis, modeling, and report writing.

## TABLE III

## LEVEL A EVALUATION

## DESIGN AND OPERATING INFORMATION

| A. Precipitator Design and Operating Data | B. Flue Gas Characterization Qualitative | C. Particulate Resistivity |
|---|---|---|
| (From observations, interviews, records) | (From interviews, or existing data) | (Measured) |
| Mechanical Design<br>Collecting Electrode System<br>Discharge Electrode System<br>Electrode Rapping Systems<br>Electrical - T/R-Systems<br>Dust Removal Systems<br>Current Mechanical and Electrical Condition<br>Current Collection Efficiency | Volumetric Flow rates<br>Qualitative data on fractional amounts of $H_2O$, $O_2$, $N_2$, CO, $CO_2$.<br>Evidence of $SO_2$, $SO_3$, $H_2SO_4$, or any other substance affecting ESP performance.<br>Inlet & Outlet Gas Temperatures | Laboratory Measurement - Grab Sample<br>In Situ Measurement - Resistivity Probe at Inlet Port |
| Refer to Section 2.1 | Refer to Section 2.5 | Refer to Section 2.4 and Appendices E and F. |

## TABLE IV

## LEVEL B EVALUATION

## PARTICULATE MASS SAMPLING & QUANTITATIVE GAS ANALYSIS

A. Pre-test Site Survey (Observation, interview, records)

Sampling Locations
Accessibility
Work Platforms
Number of Ports
Diameter of Ports
Laboratory Space
Typical Flue Gas Conditions
Typical Mass Concentrations and Particle Size Distributions*
Necessity of Substrate Conditioning

*Qualitative

Refer to Section 1 and Appendix A

B. Precipitator Inlet and Outlet Mass Sampling

Sampling Train Type
Flue Depth
Traversing Capability
Velocity/Temperature Traverse
Gas Temperature
Probe Heating
Filter Integrity
Isokinetic Sampling
Sampling Times
Entrained Water
Number of Samples
Process Variations
Rapping Puffs
Hopper Blow-off
Integrated or Time Averaged Sample

Refer to Section 2.2 and Appendices B and C

C. Quantitative Gas Analysis

ORSAT Measurement of $O_2$, $N_2$, CO, $CO_2$
EPA Methods Measurements of $SO_2$, $H_2SO_4$, $NO_x$, $SO_3$
Alternate Method for Measurement of $SO_2$ & $SO_3$
Other Gases Particular to Source Under Consideration.

Refer to Section 2.5 and Appendix G

If the process or emissions are thought to be cyclical, a decision must be made whether or not to obtain representative samples during each part of the cycle. Again, pertinent sections of this report are referenced in the table.

## 3.4 LEVEL C PRECIPITATOR EVALUATION

Level A and B tests may not yield sufficient data if the objectives of the tests include resolution of an opacity problem or space-charge problem, or if a theoretical model is to be used to compare with the test results. For each of these, it is necessary to measure the particle size distribution. Therefore, in addition to the Level B evaluation procedure, the Level C evaluation outline in Table V lists procedures for measuring the inlet and outlet particle size distribution and the precipitator fractional efficiency. Some information on rapping losses can be obtained with optical particle counters, as described in Section B.4, or by sampling with impactors during and between rapping cycles.

## TABLE V

## LEVEL C EVALUATION

## PARTICLE SIZE DISTRIBUTION MEASUREMENTS

A. Inlet
   Process Variations

B. Outlet
   Process Variations
   Rapping Puffs
   Hopper Blow-off

Measurement Methods

Size Range

0.3-20.0 µm

Cascade Impactors

| High Loading | Low Loading |
|---|---|
| Brink | Andersen |
| | MRI |
| | Sierra |
| | U. of W. |

Isokinetic Sampling
Jet Velocity Limits
Nozzle Selection
Precutter Selection
Loading Limits
Substrate Integrity
Number of Samples
Traversing
Vertical/Horizontal
Condensible Vapors
Heating/Entrained Water
Extractive Sampling
Sampling Times

Refer to Section 2.3.2 and Appendix C

0.3-10 µm

Series Cyclones

Accuracy/Resolution
Sampling Times
Back Up Filters
Filter Integrity
Filter Loading
Constant Flow rate
Entrained Water
Traversing
Isokinetic sampling
Extractive Sampling
Probe Losses

Refer to Section 2.3.2

0.3-10 µm 0.01-0.3 µm

Optical Diffusional/CNC

Extractive Sampling
Sample Conditioning
Dilution of Sample
Gas Composition
Condensible Vapors
Heated Lines
Probe Losses
Real Time Monitoring
Duct Pressures

Refer to Sections 2.3.3 & 2.3.4
and Appendix D

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# APPENDIX A

# AEROSOL FUNDAMENTALS, NOMENCLATURE, AND DEFINITIONS

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# APPENDIX A

# AEROSOL FUNDAMENTALS, NOMENCLATURE, AND DEFINITIONS

## A.1 GENERAL DISCUSSION

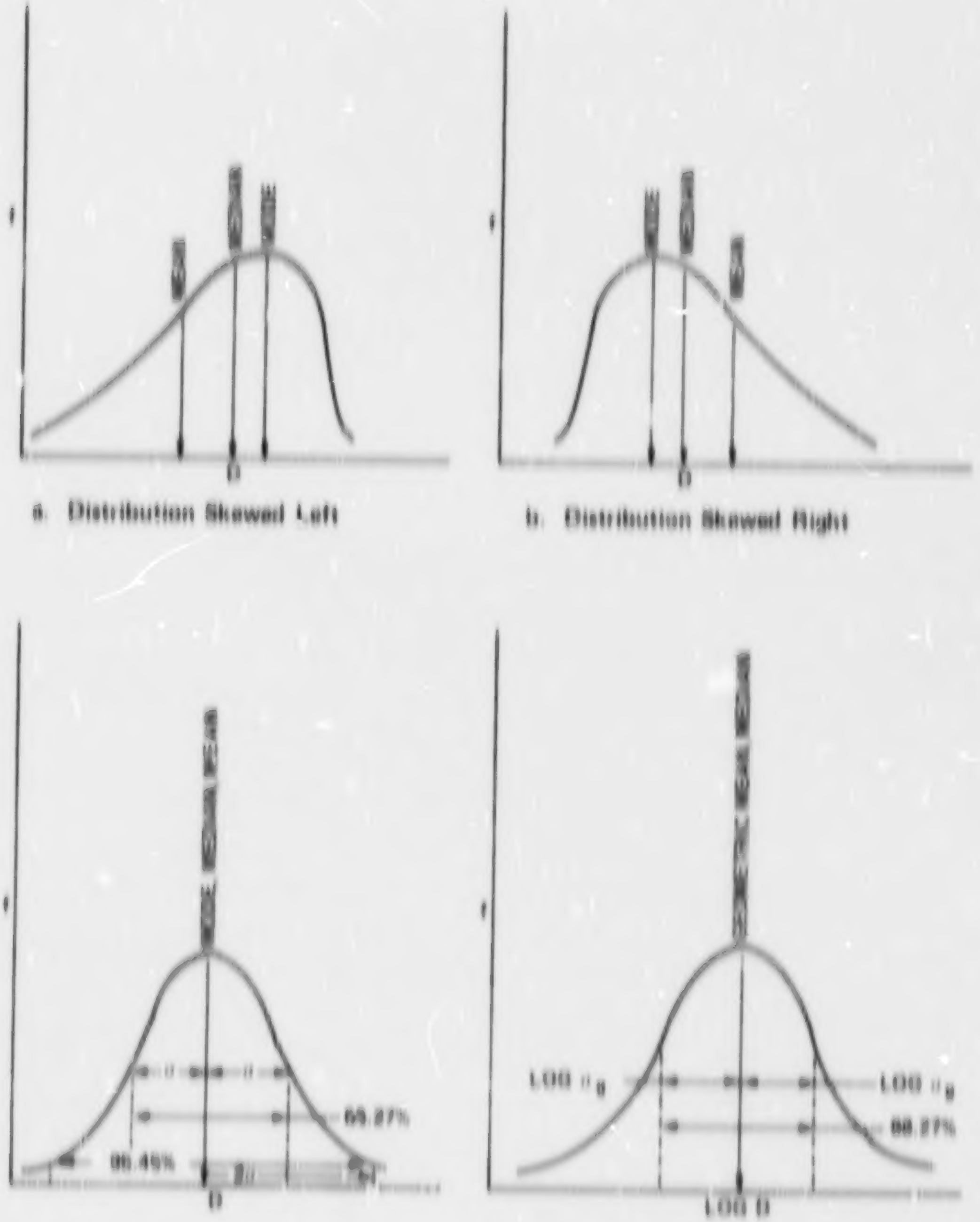
The particulate matter suspended in industrial gas streams may be in the form of nearly perfect spheres, regular crystalline forms other than spheres, irregular or random shapes, or as agglomerates made up from combinations of these. It is possible to discuss particle size in terms of the volume, surface area, projected area, projected perimeter, linear dimensions, light scattering properties, or in terms of drag forces in a liquid or gas (mobility). Particle sizing work is frequently done on a statistical basis where large numbers of particles, rather than individuals, are sampled. For this reason the particles are normally assumed to be spherical. This convention also makes transformation from one basis to another more convenient.

Experimental measurements of particle size normally cannot be made with a single instrument if the size range of interest extends over much more than a decimal order of magnitude. Presentations of size distributions covering broad ranges of sizes then must include data points which may have been obtained using different physical mechanisms. Normally the data points are converted by calculation to the same basis and put into tabular form or fitted with a histogram or smooth curve to represent the particle size distribution. Frequently used bases for particle size distributions are the relative number, volume, surface area, or mass of particles within a size range. The size range might be specified in terms of aerodynamic, Stokes, or equivalent PSL diameter. There is no standard equation for statistical distributions which can be universally applied to describe the results given by experimental particle size measurements. However, the log-normal distribution function has been found to be a fair approximation for some sources of particulate and has several features which make it convenient to use. For industrial sources the best procedure is to plot the experimental points in a convenient format and to examine the distribution in different size ranges separately, rather than trying to characterize the entire distribution by two or three parameters. The ready availability of inexpensive programmable calculators which can be used to convert from one basis to another compensates greatly for the lack of an analytical expression for the size distribution.

## A.2 PARTICLE SIZE DISTRIBUTIONS

Figure A1 shows plots of generalized unimodal particle size distributions which will be used to graphically illustrate the terms which are commonly used to characterize an aerosol. Occasionally size distribution plots exhibit more than one peak. A size distribution with two peaks would be called bimodal. Such distributions can frequently be shown to be equivalent to the sum of two or more distributions of the types shown in Figure A1. If a distribution is symmetric or bell shaped when plotted along a linear abscissa, it is called a "normal" distribution (Figure A1c). A distribution that is symmetric or bell shaped when plotted on a logarithmic abscissa is called "log-normal" (Figure A1d).

Interpretation of the frequency or relative frequency shown as f in Figure A1 is very subtle. One is tempted to interpret this as the amount of particulate of a given size. This interpretation is erroneous however and would require



a. Distribution Skewed Left

b. Distribution Skewed Right

c. Normal Distribution

d. Log Normal Distribution

*Figure A1. Examples of frequency or particle size distributions. D is the particle diameter.*

that an infinite number of particles be present. The most useful convention is to define f in such a way that the area bounded by the curve (f) and vertical lines intersecting the abscissa at any two diameters is equal to the amount of particulate in the size range indicated by the diameters selected. f then is equal to the relative amount of particulate in a narrow size range about a given diameter.

The median divides the area under the frequency curve in half. For example, the mass median diameter (MMD) of a particle size distribution is the size at which 50% of the mass consists of particles of larger diameter, and 50% of the mass consists of particles having smaller diameters. Similar definitions apply for the number median diameter (NMD) and the surface median diameter (SMD).

The term "mean" is used to denote the arithmetic mean of the distribution. In a particle size distribution the mass mean diameter is the diameter of a particle which has the average mass for the entire particle distribution. Again, similar definitions hold for the surface and number mean diameters.

The mode represents the diameter which occurs most commonly in a particle size distribution. The mode is seldom used as a descriptive term in aerosol physics.

The geometric mean diameter is the diameter of a particle which has the logarithmic mean for the size distribution. This can be expressed mathematically as:

$$\log D_g = \frac{\log D_1 + \log D_2 + \ldots \log D_N}{N} \qquad \text{(A1a)}$$

or as

$$D_g = \left(D_1 D_2 D_3 \cdot \cdot \cdot D_N\right)^{1/N} \qquad \text{(A1b)}$$

The standard deviation (σ) and relative standard deviation (s) are measures of the dispersion (spread, or polydispersity) of a set of numbers. The relative standard deviation is the standard deviation of a distribution divided by the mean, where σ and the mean are calculated on the same basis; i.e., number, mass, or surface area. A monodisperse aerosol has a standard deviation and relative standard deviation of zero. For many purposes the standard deviation is preferred because it has the same dimensions (units) as the set of interest. In the case of a normal distribution, 68.27% of the events fall within one standard deviation of the mean, 95.45% within two standard deviations, and 99.73% within three standard deviations.

### A.2.1 Cumulative and Differential Graphs

Field measurements of particle size usually yield a set of discrete data points which must be manipulated or transformed to some extent before interpretation. The resultant particle size distribution may be shown as tables, histograms, or graphs. Graphical presentations are the conventional and most convenient format and these can be of several forms.

Cumulative size distributions - Cumulative mass size distributions are formed by summing all the mass containing particles less than a certain diameter and plotting this mass versus the diameter. The ordinate is specifically equal to

$$M(j) = \sum_{t=1}^{j} M_t ,$$

where $M_t$ is the amount of mass contained in the size interval between $D_t$ and $D_{t-1}$.

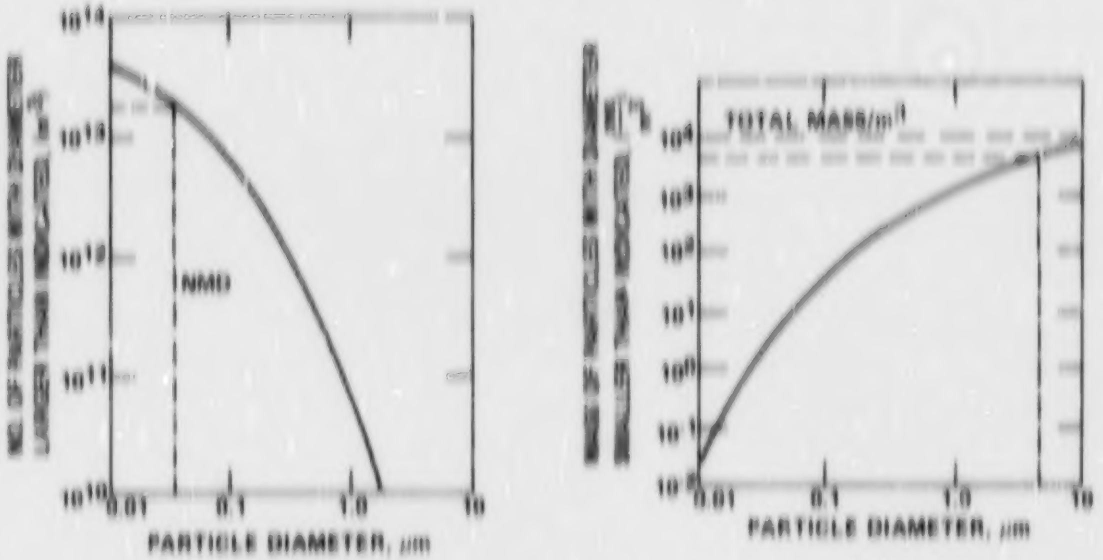
The abscissa would be equal to $D_j$. Cumulative plots can be made for surface area and number of particles per unit volume in the same manner. Examples of cumulative mass and number graphs are shown in Figures A2b and A2a, respectively, for the effluent from a coal fired power boiler. Although cumulative plots obscure some information, the median diameter and total mass per unit volume can be obtained readily from the curve. Because both the ordinate and abscissa extend over several orders of magnitude, logarithmic axes are normally used for both.

A second form of cumulative plot which is frequently used is the cumulative percent of mass, number, or surface area contained in particles having diameter smaller than a given size. In this case the ordinate would be, on a mass basis:

$$\text{Cumulative percent of mass less than size} = \frac{\sum_{t=1}^{j} M_t}{\sum_{t=1}^{N} M_t} \times 100\%. \quad \text{(A2)}$$

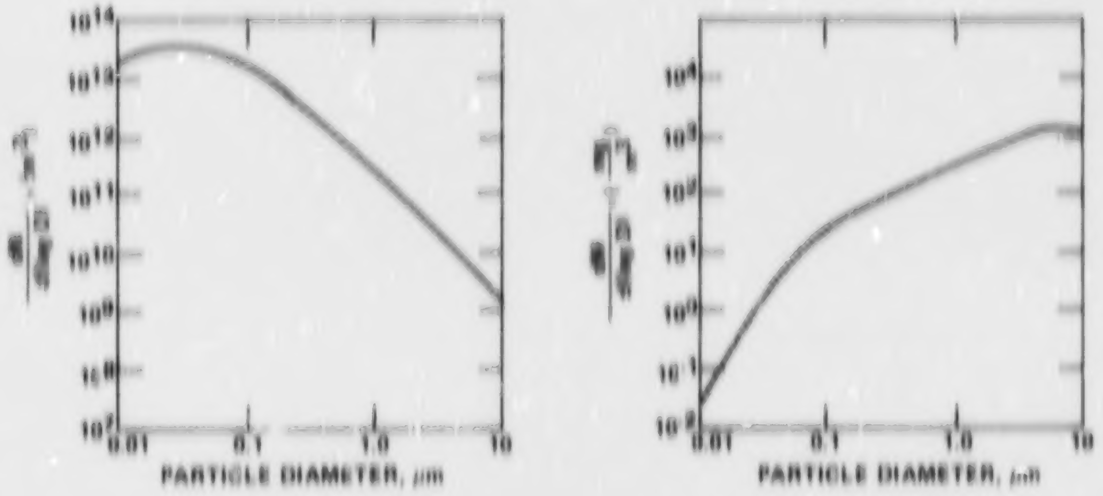
The abscissa would be log $D_j$. Special log-probability paper is used for these graphs, and for log-normal distributions the data set would lie along a straight line. For such distributions the median diameter and geometric standard deviation can be easily obtained graphically. Figures A3a and A3b show cumulative percent graphs for the size distribution shown in Figure A2a and a log-normal size distribution.

Differential size distributions - Differential particle size distribution curves are obtained from cumulative plots



a. Cumulative No. Graph

b. Cumulative Mass Graph



c. Differential No. Graph

d. Differential Mass Graph

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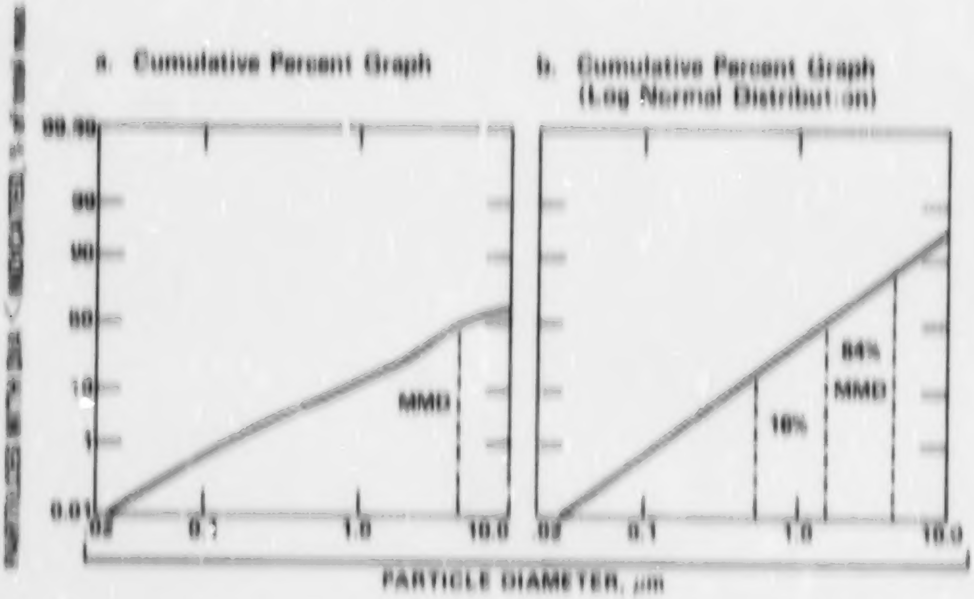
*Figure A2. A single particle size distribution presented in four ways. The measurements were made in the effluent from a coal-fired power boiler.*



a. Cumulative Percent Graph

b. Cumulative Percent Graph (Log Normal Distribution)

*Figure A3. Size distributions plotted on log probability paper.*

by taking the average slope over a small size range as the ordinate and the geometric mean diameter of the range as the abscissa. If the cumulative plot were made on logarithmic paper the frequency (slope) would be, taking finite differences:

$$\frac{\Delta M}{\Delta(\log D)} = \frac{M_j - M_{j-1}}{\log D_j - \log D_{j-1}} , \qquad \text{(A3)}$$

and the abscissa would be $D_G = \sqrt{D_j D_{j-1}}$ where the size range of interest is bounded by $D_j$ and $D_{j-1}$. $M_j$ and $M_{j-1}$ correspond to the cumulative masses below these sizes. Differential number and surface area distributions can be obtained from cumulative graphs in precisely this same way. Differential graphs show visually the size range where the particles are concentrated with respect to the parameter of interest. The area under the curve in any size range is equal to the amount of mass (number, or surface area) consisting of particles in that range, and the total area under the curve corresponds to the entire mass (number, or surface area) of particulate in a unit volume. Again, because of the extent in particle size and the emphasis on the fine particle fraction, these plots are normally made on logarithmic scales. Figures A2c and A2d are examples of differential graphs of particle size distributions.

Log-normal size distributions - The formation of aerosols by different means frequently result in particle size distributions which obey the log-normal law. For log-normal particle size distributions the geometric mean and median diameters coincide.

The normal distribution law is, on a mass basis:

$$f = \frac{dM}{dD} = \frac{1}{\sigma\sqrt{2\pi}} \exp\left[-\frac{(D-D_m)^2}{2\sigma^2}\right] , \qquad (A4)$$

The log-normal distribution law is derived from this equation by the transformation $D \rightarrow \log D$

$$f = \frac{dM}{d(\log D)} = \frac{1}{\log \sigma_g \sqrt{2\pi}} \exp\left[-\tfrac{1}{2}\left(\frac{\log D - \log D_{gm}}{\log \sigma_g}\right)^2\right] \qquad (A5)$$

where $\sigma_g$, the geometric standard deviation, is obtained by using the transformation $D \rightarrow \log D$ in equation A4. This distribution is symmetric when plotted along a logarithmic abscissa and has the feature that 68.3% of the distribution lies within one geometric standard deviation of the geometric mean on such a plot. Mathematically, this implies that $\log \sigma_g = \log D_{84.14} - \log D_g$ or $\log D_g - \log D_{15.86}$ where $D_{84.14}$ is the diameter below which 84.14% of the distribution is found, etc. This can be simplified to yield:

$$\sigma_g = \frac{D_{84}}{D_g} , \qquad (A6)$$

$$\sigma_g = \frac{D_g}{D_{16}} , \text{ or} \qquad (A7)$$

$$\sigma_g = \left(\frac{D_{84}}{D_{16}}\right)^{\frac{1}{2}} \qquad (A8)$$

When plotted on log-probability paper, the log-normal distribution is a straight line on any basis and is determined completely by the knowledge of $D_g$ and $\sigma_g$. This is illustrated in Figure A3b. Another important feature is the relatively simple relationships among log-normal distributions of different bases. If $D_{gm}$, $D_{gs}$, $D_{gvs}$, and $D_{gN}$ are the geometric mean diameter of the mass, surface area, volume-surface, and number distribution, then:

$$\log D_{gs} = \log D_{gm} - 4.6 \log^2 \sigma_g, \qquad (A9)$$

$$\log D_{gvs} = \log D_{gm} - 1.151 \log^2 \sigma_g, \text{ and} \qquad (A10)$$

$$\log D_{gM} = \log D_{gm} - 6.9 \log^2 \sigma_g. \qquad (A11)$$

The geometric standard deviation remains the same for all bases.

More examples of particle size distribution graphs are given in the data reduction sections of Appendices C and D. The following section in this Appendix lists useful definitions, equations, and nomenclature for aerosol sampling.

## A.3 NOMENCLATURE AND DEFINITIONS

### A.3.1 Definition of Particle Diameter

Aerodynamic diameter, $D_A$ - The aerodynamic diameter of a particle is the diameter of a sphere of unit density which has the same settling velocity in the gas as the particle of interest.

Aerodynamic impaction diameter, $D_{AI}$ - The $D_{AI}$ of a particle is an indicator of the way that a particle behaves in an inertial impactor or in a control device where inertial impaction is the primary mechanism for collection. If the particle Stokes diameter is known, $D_s$, the $D_{AI}$ is equal to:

$$D_{AI} = D_s\sqrt{\rho C}\ , \qquad (A12)$$

where $\rho$ is the particle density, $gm/cm^3$, and
$C$ is the slip correction factor.

Stokes diameter, $D_s$ - If the density of a particle is known, the Stokes diameter may be used to describe particle size. This is the diameter of a sphere having the same density which behaves aerodynamically as the particle of interest. For spherical particles, the Stokes number is equal to the actual dimensions of the particle.

An average density for the particles can be obtained from volume-weight data using a helium pycnometer if large enough samples are available. The validity of size information based on an average density depends upon the uniformity of the density from particle to particle, particularly with respect to size. Visual inspection of some size-classified samples from flue gases sometimes shows a variation in color with size which would seem to indicate compositional inhomogeneities.

Equivalent polystyrene latex (PSL) diameter - The intensity of light scattered by a particle at any given angle is dependent upon the particle size, shape, and index of refraction. It is impractical to measure each of these parameters and the theory for irregularly shaped particles is not well developed. Sizes based on light scattering by single particles are therefore usually estimated by comparison of the intensity of scattered light from the particle with the intensities due to a series of calibration spheres of very precisely known size. Most commonly these are PSL spheres.* Spinning disc and vibrating orifice aerosol generators can be used to generate monodisperse calibration aerosols of different physical properties. Because most manufacturers of optical particle sizing instruments use PSL spheres to calibrate their instruments, it is convenient to define an equivalent PSL diameter as the diameter of a PSL sphere which gives the same response with a particular optical instrument as the particle of interest.

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*Available from The Dow Chemical Company, P. O. Box 68511, Indianapolis, Indiana 46268.

Equivalent volume diameter - Certain instruments, such as the Coulter Counter, have, as the measured size parameter, the volumes of the individual particles. Size distributions from such techniques are given in terms of spheres having the same volume as the particles of interest.

### A.1.2 Mean Free Path of Gas

The mean free path of a gas, which is the average distance that molecules travel between collisions, is an important parameter in determining the aerodynamic behavior of particles. For practical purposes, the mean free path is given with sufficient accuracy by the following equation.

$$\lambda = \frac{2\mu}{1.01\text{x}10^6 P} \left(\frac{8.3\text{x}10^7 T}{8\ MM}\right)^{\frac{1}{2}} \qquad \text{(A13)}$$

where $\mu$ is the viscosity of the gas, poise,
$P$ is the pressure of the gas, atm,
$T$ is the temperature, °Kelvin, and
$MM$ is the mean molecular weight.

### A.1.3 Slip Correction Factor

Stokes law can be applied to submicron particles if a correction factor, C, is used.

$$C = 1 + \frac{2\lambda}{D} \; 1.23 + 0.41 \exp(\frac{-0.44D}{\lambda}) \; , \qquad (A14)$$

where $\lambda$ is the mean free path of the gas, μm, and
$D$ is the particle diameter, μm.

The constants in equation A14 were determined empirically for air at standard temperature and pressure, and are thus only approximate for stack conditions. If the exponential term is neglected, equation 14 is referred to as the Cunningham correction factor.

### A.3.4 Viscosity of Gas

A parameter which appears in many equations describing flow fields, drag forces, and shear forces in liquids and gases is the viscosity.

In order to find the viscosity of the flue gas, $\mu$, the viscosity of the pure gas components of the flue gas must first be found. Viscosity is a function of temperature, and the temperature difference in different flue gases can be quite significant. The following equations (derived from curves fitted to viscosity data from the Handbook of Chemistry and Physics, Chemical Rubber Company Publisher, 54 Edition, 1973-1974, pp. F52-55), are used to find the viscosities of $CO_2$ ($\mu_1$), CO($\mu_2$), $N_2$ ($\mu_3$), $O_2$($\mu_4$) and $H_2O$($\mu_5$).

$$\mu_1 = 138.494 + 0.499T - 0.267 \times 10^{-3}T^2 + 0.972 \times 10^{-7}T^3$$
$$\mu_2 = 165.763 + 0.442T - 0.213 \times 10^{-3}T^2$$
$$\mu_3 = 167.086 + 0.417T - 0.139 \times 10^{-3}T^2$$
$$\mu_4 = 190.187 + 0.558T - 0.336 \times 10^{-3}T^2 + 0.139 \times 10^{-6}T^3$$
$$\mu_5 = 87.800 + 0.374T + 0.238 \times 10^{-3}T^2$$

where T is the temperature of the flue gas in degrees Celsius.

The units of $\mu$ are $10^{-6}$ g/cm-sec. Next, these values of $\mu_1$ through $\mu_5$ are used in a general viscosity equation for a mixture of any number of components (See "A Viscosity Equation for Gas Mixtures" by C. R. Wilke, Journal of Chemical Physics, Volume 8, Number 4, April 1950, page 517) used to find the viscosity of the flue gas:

$$\mu = \sum_{i=1}^{n} \frac{\mu_i}{\left[1 + \frac{1}{X_i} \sum_{\substack{j=1 \\ j \neq i}}^{j=n} X_j \phi_{ij}\right]} \qquad \text{(A15)}$$

where $\phi_{ij}$ is given by the equation:

$$\phi_{ij} = \frac{\left[1 + (\mu_i/\mu_j)^{1/2} (M_j/M_i)^{1/4}\right]^2}{(4/\sqrt{2}) \left[1 + (M_i/M_j)\right]^{1/2}} , \qquad \text{(A16)}$$

and $M$ = molecular weight of a component in the mixture,
$X$ = mole fraction of a component in the mixture,
$\mu$ = viscosity, g/cm-sec; $\mu_1$, $\mu_2$, etc., refer to the pure components at the temperature and pressure of mixture, $\mu$ is the viscosity of the mixture, and
$\phi$ = dimensionless constant defined above.

### A.3.5 Particle-Gas Interactions

Particle relaxation time - For the purposes of this document, the particle relaxation time, $\tau$, may be defined as the time required for a particle to accelerate from some initial velocity to the velocity of the carrier gas.

$$\tau = \frac{\rho_p D^2 C}{18 \mu} \qquad \text{(A17)}$$

where $\rho_p$ is the particle density, gm/cm³,
$D$ is the particle Stokes diameter, cm,
$C$ is the slip correction factor, and
$\mu$ is the gas viscosity, poise.

Particle stopping distance - The particle stopping distance, $\ell$, is the distance travelled by a particle as it decelerates from the initial velocity of the gas to zero velocity.

$$\ell = \tau V \qquad \text{(A18)}$$

where $\tau$ is the relaxation time (sec), and
$V$ is the initial velocity of the particle (cm/sec).

Stokes number - The Stokes number, $\psi$, is the ratio of the particle stopping distance and some characteristic dimension of the sampling system. For example, if the stopping distance for particles of a given diameter is much smaller than the diameter of a sampling nozzle, ($\psi << 1$) the particles will be sampled accurately in spite of flow disturbances due to the nozzle design or sampling velocity. If the particle stopping distance is comparable in magnitude to the nozzle diameter; however, the particles may cross flow streamlines and either enter or miss the nozzle in quantities which are not proportional to the sample flowrate. Thus, for $\psi$ on the order of 0.1 or greater, isokinetic sampling is required.

Particle mobility - The ratio of the velocity of a particle to the force causing steady motion is called the mobility, b,

$$b = \frac{C}{3\pi\mu D} , \qquad \text{(A19)}$$

where $\mu$ is the gas viscosity, poise,
$D$ is the particle diameter, cm, and
$C$ is the slip correction factor.

### A.3.6 Cascade Impactor Terminology

Blank -- A blank usually refers to a controlled test run in which the particles are removed by a prefilter. If the measured stage weight is found to change significantly and consistently, the normal runs should be corrected for background.

Bounce -- Bounce in this document refers to inadequate retention of particles which strike the impaction surface. If the particle does not adhere, it is said to bounce.

Condensation -- Condensation in an impactor refers to the coalescence of vapors either into liquid particulate in the gas stream or on the impactor walls.

Control -- A control run is a technique which is used to confirm that variables are isolated. The control is made up to be as similar as possible to an actual run, but it is not run through the test situation. The control is then examined as would an actual test run. If the experimental variable changed significantly, the experiment is not properly set up.

Cut-point -- The cut-point of an impactor stage is the particle diameter for which all particles of equal or greater diameter are captured and all particles with smaller diameters are not captured. No real impactor actually has a sharp cut-point, but the $D_{50}$ of a stage is often called its cut-point.

$\underline{D_{50}}$ -- the $D_{50}$ of an impactor stage is the particle diameter at which the device is 50 percent efficient. Fifty percent of the particles of that diameter are captured and 50% are not.

The impactor cut-point, or $D_{50}$ is given by:

$$D_{50} = \sqrt{\frac{18\ \psi\ \mu\ D_j}{C\ \rho_p V_j}} \qquad \text{(A20)}$$

where $\psi$ = Stokes inertial impaction parameter, determined by calibration, dimensionless,
$\mu$ = gas viscosity, poise,
$D_j$ = impactor jet diameter (for slot impactors, the slot width, cm),
$V_j$ = gas velocity through impactor jet, cm/sec,
$C$ = slip correction factor, dimensionless,
$\rho_p$ = particle density, g/cm$^3$.

$D_{50}$(AI), aerodynamic impaction diameter, is found by setting $C$ and $\rho_p = 1.0$.
$D_{50}$(A), the aerodynamic diameter, is found by setting $\rho_p = 1.0$, and
$D_{50}$(S), Stokes diameter, is found by setting $\rho_p$ = the actual particle density.

Grease -- In impactor terminology, grease is a substance which is placed on an impactor stage or substrate to serve as an adhesive.

Isokinetic sampling -- This is sampling with the bulk fluid velocity through the impactor nozzle equal to the velocity in the duct. This is necessary to prevent sample bias.

Preconditioning -- Unwanted weight changes of impactor glass fiber collection substrates may be reduced by placing a large number of substrates inside the duct to be sampled, and pumping filtered flue gas through them for several hours. Such a procedure is referred to as "preconditioning" the substrates.

Precutter or precollector -- A collection device, often a cyclone, which is put ahead of the impactor in order to reduce the first stage loading. This is necessary in some streams because the high loading of large particulate would overload the first stage before an acceptable sample had been gathered on the last stages.

Re-entrainment -- Re-entrainment in an impactor is the phenomenon of particles which impacted on a given stage being picked up by the gas stream and moving downstream to another stage.

Stage -- A stage of an impactor is usually considered to be the accelerating jet (or plate containing multiple jets) and the surface on which the accelerated particles impact.

Substrate -- The removable, often disposable, surface on which impacted particles are collected. Substrates are characteristically light and can be weighed on a microbalance.

Wall Losses - Wall losses are that portion of the particles in the gas stream which impact with and adhere to surfaces in the impactor other than the substrates. They should be collected if possible and assigned to the proper stage catch.

# APPENDIX B
## PARTICULATE MASS CONCENTRATION MEASUREMENTS

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# APPENDIX B

## PARTICULATE MASS CONCENTRATION MEASUREMENTS

This appendix contains the details for conducting particulate mass concentration measurements using the EPA Reference Method 5 procedure (See Appendix G). The information is presented in four sections. Section B1 describes the equipment used in conducting these tests. Section B2 is concerned with preliminary procedures prior to sampling. Section B3 details the actual mass sampling procedures. Section B4 deals with data analysis after testing has been concluded. The sampling system described is depicted in Figure 13 of Section 2.2.2.

## B.1 GENERAL SAMPLING EQUIPMENT

### B.1.1 Temperature Measurement

Several temperature measurements are required in conducting a test for particulate mass loading, including the temperature of the stack gas, the particulate filter, and the cooled sample stream. The relative errors encountered in temperature measurements are usually small since absolute temperatures are used in all gas law calculations. In source testing, dial thermometers and thermocouples are usually used for making temperature measurements.

Two common scales are used in temperature measurement, the Celcius scale and the absolute Kelvin scale. Conversions for these are shown below for °C to °K and °F to °C.

$$°K = °C + 273°$$

$$°C = 5/9(°F - 32)$$

Mercury bulb thermometers - The mercury bulb thermometer operates by measurement of mercury expansion with temperature increase. This expansion is linear over the range of the temperature scale. Glass mercury thermometers break easily and this is a risk in source sampling.

Dial thermometers - Two types of dial thermometers are available for use in sampling. One is bimetallic and the other is a gas bulb thermometer. The bimetallic thermometer contains a strip of two different metals bonded together. Because of the different thermal expansion coefficients of the two metals, the bonded strip will deform with temperature, and depending on the configuration of the strip, this deformation will be transferred to a dial movement which contains a temperature scale.

Gas bulb thermometers rely on the expansion of an inert gas with temperature. This expansion is sensed as a change in pressure. The dial temperature scale is actually a pressure scale. Gas bulb thermometers are used for lower temperature ranges.

Thermocouples - Thermocouples are the most popular device for measuring high temperatures. These consist of two dissimilar wires welded together at one junction (See Figure B1). These two wires are joined to a third wire held at a reference temperature. The difference between the temperature in question and the reference junction temperature causes an electromotive force in the system which can be sensed by a potentiometer. Several metal pair types are available. Generally Chromel/Alumel will be the best choice due to resistance to oxidation. This pair is useful in the range

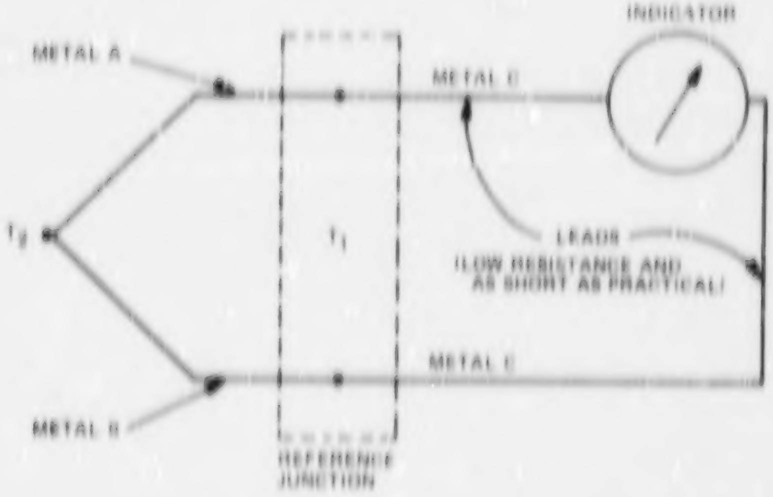

INDICATOR
METAL A
METAL C
$T_2$
$T_1$
LEADS
(LOW RESISTANCE AND
AS SHORT AS PRACTICAL)
METAL C
METAL B
REFERENCE
JUNCTION

*Figure B1. Thermocouple junction.*

from -184°C to 1260°C. Other common pairs are Copper/Constantan (-184°C to 350°C), Iron/Constantan (-158°C to 1010°C), and Platinum/Platinum 10% Rh (0°C to 1538°C).

### B.1.2 Pressure Measurement

Pressure is defined as a force per unit area. Most pressure measurements are made with local atmospheric pressure as reference. The pressure above atmospheric is considered positive, and that below negative. The absolute pressure at a point is the atmospheric plus the pressure differential.

An easy way to measure a low pressure is to balance a column of liquid against the pressure. The magnitude of the pressure can be calculated based on the measured height of the liquid column. Devices which do this are called manometers. In source sampling, manometers are often used for the determination of the stack gas velocity and the sample train flow rate. For small pressure differentials, the manometer is often inclined to increase the sensitivity. The inclined manometer is used to measure the stack velocity pressure and sample stream orifice pressure differential. It is advantageous to use manometers which have some means of protection against accidental blow out.

Mechanical pressure gauges are also available to measure low differential pressures commonly encountered as velocity pressure and orifice meter heads in sampling systems. The Magnehelic gauge manufactured by F. W. Dwyer, Mfg. in Michigan City, Indiana is an example of such an instrument. Inclined manometers, however, are generally more reliable and easier to use. They are also easier to repair.

## B.1.3 Velocity Measurement

The measurement of velocity in a duct utilizes one of the simplest devices in most sampling systems, the pitot tube. These devices are necessary because it is impossible to determine the total volumetric flow through large ducts. Only by measuring the velocity at many points and knowing the area of the duct can an accurate determination of duct volumetric flow be made. The pitot tube will not directly measure the average duct velocity but measure only the instantaneous velocity at the point at which it is located.

Several configurations are possible for pitot tubes. One, the Prandtl type is shown in Figure B2. The static pressure is measured at point W. The velocity pressure is measured at point P. The velocity, $V_s$, then is given by

$$V_s = C_p \left[\frac{2(P_p - P_w)}{\rho_s}\right]^{1/2}$$

where $V_s$ is the gas velocity, cm/sec,

$P_p$ is the measured velocity pressure, mm Hg,

$P_w$ is the measured static pressure, mm Hg,

$C_p$ is the pitot coefficient, dimensionless,

$\rho_s$ is the density of the stack gas, gm/cm$^3$.

This pitot tube shown in this figure is usually called a standard pitot tube; the $C_p$ value for this configuration is approximately 0.99.

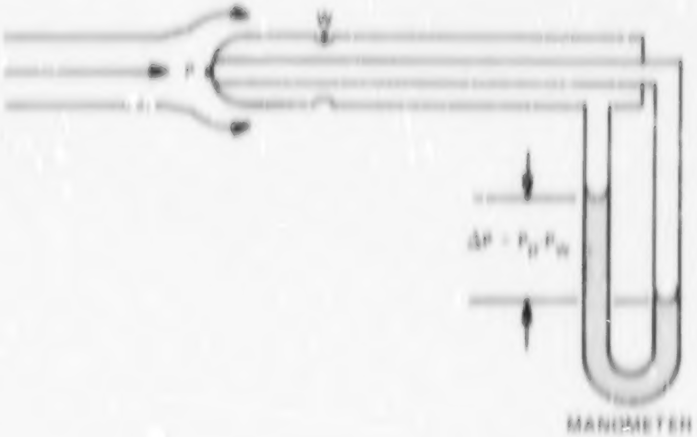

W
P
$\Delta P = P_0 - P_W$
MANOMETER

*Figure B2. Standard pitot tube.*

One configuration which does not resemble the standard pitot is the reversed or Stausscheibe (S-type) pitot tube. The S-type pitot tube is used primarily for stack testing because of one major advantage: it will not rapidly clog in gases with heavy dust concentrations. An example of one of these is shown in Figure B3. The S-type does not give the same velocity pressure as the standard pitot tube. The observed delta P is larger for a given velocity because the rear part of the tube faces downstream. This $P_s$ is a wake pressure which is lower than the static pressure. When used with a water manometer, the S-type pitot tube equation becomes

$$V_s = 422.67\ C_p \left(\frac{T_s \Delta P}{P_s M_s}\right)^{\frac{1}{2}}$$

where $V_s$ is the gas velocity, cm/sec,

$\Delta P$ is the velocity head ($P_p - P_s$), mm Hg

$T_s$ is the stack gas temperature, °K,

$P_s$ is the absolute stack gas pressure, mm Hg,

$M_s$ is the molecular weight of the stack gas, gm/gm-mole, and

$C_p$ is the pitot tube correction factor.

For the limits of 0.025 to 25.4 cm of water velocity pressure, $C_p$ for a standard (Prandtl) pitot tube usually takes on values of 0.98 to 1.00. The $C_p$ of an S-type pitot tube usually is between 0.83 and 0.87. Each must be calibrated before a test, preferably in a gas stream in which the gas properties and velocity are similar to those of the test conditions.

The calibration of a pitot tube requires a gas stream of constant and known velocity. Thus, a wind tunnel facility

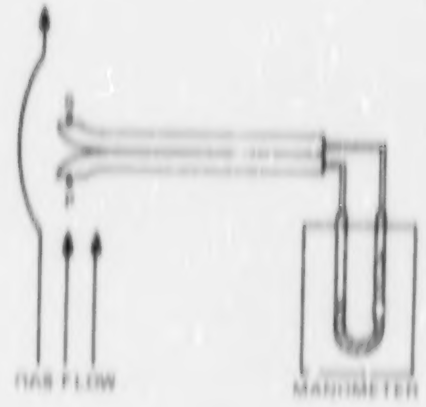

GAS FLOW
MANOMETER
3830 068

*Figure B3   S-type pitot tube.*

should be available. However, the S-type pitot tube can be calibrated against the standard pitot tube. This is the procedure designated in EPA Reference Method 2.

The following equation applies in this case:

$$C_{p_{test}} = C_{p_{std}} \frac{\Delta P_{std}}{\Delta P_{test}}$$

where $C_{p_{test}}$ = Coefficient of the S-type pitot tube,

$C_{p_{std}}$ = Coefficient of the standard pitot tube,

$\Delta p_{test}$ = Velocity pressure measured by the S-type pitot tube, and

$\Delta p_{std}$ = Velocity pressure measured by the standard pitot tube.

To calibrate the S-type pitot tube, the velocity pressure is measured at the same point with both the S-type and standard pitot tubes. Both pitot tubes must be properly aligned in the flow field. The appropriate values are inserted in the above equation and the coefficient for the S-type pitot tube is calculated. If $C_{p_{std}}$ is not known, then a value of 0.99 should be used.

The coefficients for the S-type pitot tube should be determined first with one leg, then with the other leg pointed downstream. If the computed coefficients differ by more than 0.01, the pitot tube should not be used without proper labeling.

The determination of the average stack gas velocity, $\bar{v}_s$, is one of the greatest sources of error in stack sampling. Therefore, it is recommended that the pitot tube be recalibrated on a regular basis.

At extremely low or high velocities the pitot method is inaccurate and unreliable. There are several other mechanical and electronic methods which are available, hot wire anemometers, rotating vane anemometers and certain fluidic devices.

### B.1.4 Nozzles

The nozzle is considered the initial sampling system boundary. It removes a portion of the effluent from the duct and delivers it to the sampling probe. The nozzle has several restrictions in its use:

1. It should not disturb the duct gas stream flow.
2. It should not alter the particulate being sampled.
3. It should not add to the sample being collected.
4. It should be of a size allowing easy access.

For particulate sampling, the nozzle should disturb the gas flow as little as possible or the sample will not be representative. Any nozzle will disturb the flow, but a thin wall, sharp edged nozzle disturbs it the least. In the case of partiuulates, any bends in the nozzle will cause impingement of larger particles. The nozzle must then be cleaned carefully and any material found within it must be added to the total collected particulate.

### B.1.5 Probes

The probe is the sampling interface between the gas stream in the duct and the external sampling train (See Figure B4). It is exposed internally and externally to the flue gas at the nozzle end and ambient air at the exit end. The probe should not alter the sample in any way. It must be able to support itself. It must be easy to clean and it must not add to the sample. Ideally the sample should

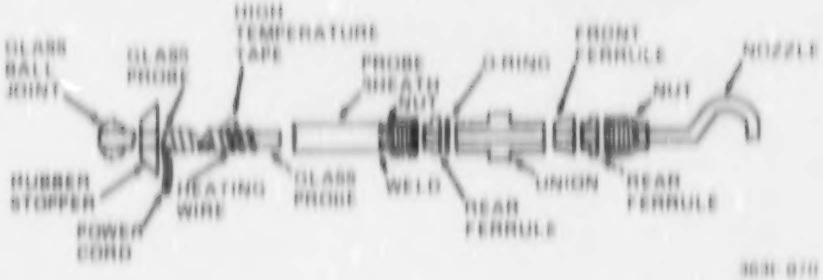

GLASS BALL JOINT
GLASS PROBE
HIGH TEMPERATURE TAPE
PROBE SHEATH
NUT
O-RING
FRONT FERRULE
NUT
NOZZLE
RUBBER STOPPER
POWER CORD
HEATING WIRE
GLASS PROBE
WELD
REAR FERRULE
UNION
REAR FERRULE
3631 070

*Figure B4. Probe and nozzle assembly.*

be delivered to the sample train at the stack temperature. In most instances this requires that the probe be heated to maintain the sample at stack conditions and prevent condensation. Structurally the probe must support itself, the nozzle, the pitot tube, and sometimes a thermocouple. This strength is obtained by use of a metal sheath around the probe. The requirement of probe cleaning conflicts with the structural needs. The surface of a glass tube is much more easily cleaned than a metal tube, but it is more fragile. In most cases glass probes over 2 meters in length are impractical. If a metal tube is used, some material may become trapped in the rough surface of the probe.

In the case of extremely high temperatures, the only practical choice is to use a water-cooled, high quality, stainless steel probe.

Glass has another advantage which should be considered. It is for all practical purposes chemically inert. This is not true of stainless steel especially if there are acid gases present in the gas stream which is being sampled.

### B.1.6 Gaseous Sample Collectors

There are four main types of gas sample collection devices. One type is the cold trap which condenses vapors in the sample flow stream. Another type is that which contains a solid adsorbent. This removes the gas from the stream by surface adsorption. The third type is the grab sample container. The fourth uses the principle of gas absorption by a liquid.

Gas absorbers are generally called impingers or bubblers. Their efficiency depends on the diffusivity of the gases, the retention time in the devices, the bubble size, and the gas solubility. There are four general types of impingers; the midget impinger, the fritted glass bubbler, the modified impinger tip bubbler, and the most commonly used type, the Greenburg-Smith impinger.

### B.1.7 Particulate Sample Collectors

Filtration is the basic method for particulate collection. There are three major types of filters available today: the flat glass fiber filter, the ceramic Alundum filter, and the glass fiber bag.

Alundum thimbles are subject to variations in their particle retention efficiency as they are used because of changes in their porosity as a particulate cake forms on them. Therefore new filters should not be used when testing relatively clean gas streams. In addition, when the thimble is used in relatively clean gas streams, only small amounts of particulate may be collected and weighing accuracy suffers. There are sometimes problems with unknown penetration characteristics of the glass fiber bags and flat filters. In many sampling trains a small cyclone precollector is used to remove larger particulate and allow longer sampling times.

### B.1.8 Sample Flow Rate Meters

The sharp edged orifice meter is a simple and accurate method to measure instantaneous volumetric flow rate. In source sampling it is used in conjunction with a total volume gas meter. As the gas passes through the orifice restriction, a pressure drop is created. The following equation is used for determining the flow rate through an orifice.

$$Q_m = K_m \left( \frac{T_m \Delta P_{om}}{P_m \, M_m} \right)^{1/2}$$

where $Q_m$ = Gas flow rate, cm³/sec,

$K_m$ = Proportionality factor determined by calibration,

$T_m$ = Upstream gas temperature, °K,

$\Delta P_{om}$ = Orifice meter pressure drop, mm $H_2O$,

$P_m$ = Upstream absolute pressure, mm Hg, and

$M_m$ = Molecular weight of gas, gm/gm-mole.

For a given orifice, $K_m$ must be determined by calibration. $K_m$ is a function of Reynolds number and thus will not be a constant over the entire range of flow rates. However for a small range of $Q_m$, such as for most sampling cases, $K_m$ is a constant. Thus it is important to calibrate the orifice for the range of flow rates anticipated. Generally commercial sampling trains have orifices with delta $P_{om}$ of 0-25 cm $H_2O$ over the useful flow rate range. For calibration, see Section 8.1.9.

## 8.1.9 Total Sample Volume

The total volume of gas sampled must be determined in most sampling trains. This provides the volume necessary to calculate the particulate concentration. Dry gas meters with capacities from 0.094-10.8 liters/sec (0.2 to 150 ft³/min) are generally used. For sampling, the smallest dial face division should be 5 cm³ (0.01 ft³) because the meter movement is not smooth over one revolution. The dry gas meter is calibrated using a wet test meter. In a wet test meter the gas displaces water in a chamber and causes the rotor to revolve. It should be noted that the gas leaving the wet test meter is saturated with water vapor. The setup for calibrating a dry gas meter and sampling orifice is shown in Figure B5. The pertinent information should be entered on a form similar to that shown in Figure B6. The following equation is used to determine how well the dry gas meter performs as compared with the wet test meter. If the ratio defined by the equation is less than 0.99 or greater than 1.01, then the dry gas meter should be readjusted and recalibrated.

$$\gamma = \frac{V_w P_b (T_d)}{V_d (P_b + \Delta M) (T_w)}$$

where $\gamma$ is the ratio of accuracy of the wet test meter to the dry gas meter,

$V_s$ is the gas volume passing through the wet test meter, $cm^3$,

$P_b$ is the absolute barometric pressure, mm Hg,

$T_d$ is the average temperature of the gas in the dry gas meter, °K,

$T_w$ is the temperature of the gas in the wet test meter, °K,

$\Delta M$ is the orifice meter pressure drop, mm Hg,

$V_d$ is the volume of gas passing through the dry gas meter, $cm^3$, and

$\theta$ is the time, seconds, for sampling the gas volume, $V_w$.

The data in Figure B6 can be used to calibrate the orifice meter. In making these calculations the wet test meter is used for the flow rate, $Q_m$, and

$$Q_m = \frac{V_w}{\theta} .$$

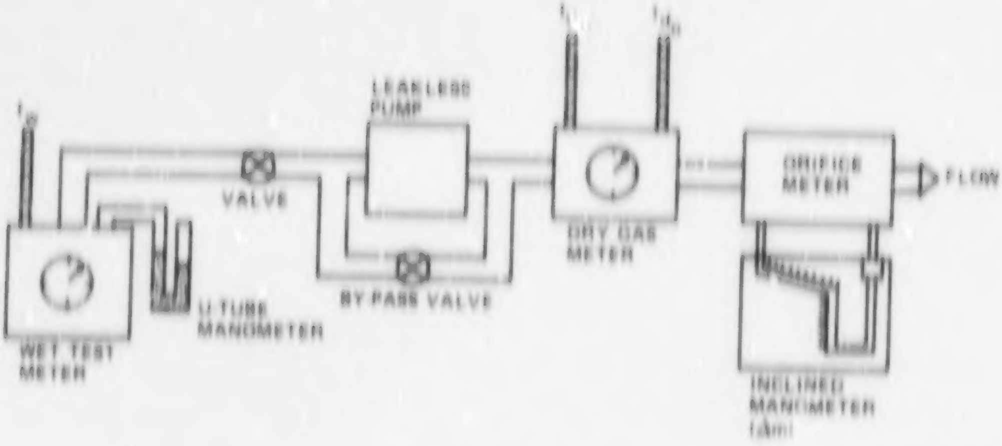

$t_w$
$t_{d_i}$
$t_{d_o}$
LEAKLESS PUMP
VALVE
DRY GAS METER
ORIFICE METER
FLOW
BY-PASS VALVE
U-TUBE MANOMETER
WET TEST METER
INCLINED MANOMETER
(ΔH)

*Figure B5. Set-up for calibration of dry gas meter and orifice meter.*

DRY GAS METER NO. ______________ ORIFICE METER NO. ______________

DATE ______________ WET TEST METER NO. ______________

BAROMETRIC PRESSURE $P_b$ ______ mm Hg CALIBRATED BY ______________

| ORIFICE SETTING $\Delta H$ mm Hg | WET TEST METER VOLUME $V_w$ $cm^3$ | DRY TEST METER VOLUME $V_d$ $cm^3$ | TEMPERATURE | | | | TIME $\theta$ sec | $\gamma$ | $K_m$ |
|---|---|---|---|---|---|---|---|---|---|
| | | | WET TEST | DRY TEST | | | | | |
| | | | $t_w$ °K | $t_{d_i}$ °K | $t_{d_o}$ °K | $\bar{t}_d$ °K | | | |
| 0.5 | | | | | | | | | |
| 1.0 | | | | | | | | | |
| 1.5 | | | | | | | | | |
| 2.0 | | | | | | | | | |
| 2.5 | | | | | | | | | |
| 3.0 | | | | | | | | | |
| 3.5 | | | | | | | | | |
| 4.0 | | | | | | | | | |
| | | | | | | | AVERAGE ➡ | | |

[illegible]

Figure B6. Data sheet for calibrating dry gas meter and orifice meter.

## A.1.10 Gas Conditioning

Often the gas sample must be conditioned or treated before or while it is passed through the sampling train components. This is done either to preserve the sample or to prevent damage to the sampling train. Typical gas conditioning operations include condensing, drying, heating, and dilution.

### Condensing

Condensers are used to remove water and other vapors from the gas sample. They work on the prinicipIe that the partial pressure of water vapor decreases with a decrease in sample temperature. For example, as indicated in steam tables, the partial pressure of water vapor at 0°C is only 0.15% of the partial pressure at 150°C. Thus the use of an ice bath type condenser is an effective way to remove water vapor from a gas sample. This provides the moisture content which must be determined in order to calculate the molecular weight of the stack gas.

The ice bath condenser is also used in source sampling to protect other components from damage. The deposition of water vapor and water soluble constituents in such components as the dry gas meter and pump can cause severe damage.

The ice bath condenser usually consists of several wet and dry impingers connected in series, but it may be as simple as a piece of coiled tubing. A measured initial amount of water is put into the impinger-type condenser to assist in the condensation process. When the sampling train is operated a known amount of sample gas is passed through the system.

By observing the pressure and temperature operating conditions, the amount of water condensed, and the amount of gas passing through the system, the moisture content of the gas stream can be calculated.

## Drying

In source sampling trains, gas drying is used to accomplish the same objectives as condensing. The drying operation is achieved using special chemicals which have a great affinity for water vapor. One such chemical is silica gel. The silica gel strongly adsorbs water and hence its change in weight can be used to calculate the moisture content of the gas stream. Indicating silica gel, which is granular and has a bright blue color, can be obtained commercially. As it becomes saturated with water vapor, its color changes to a light pink. If this method is to be used to determine the moisture content of a gas sample, care must be taken to insure that all particulate matter is removed first and there is no other major constituent in the gas stream which may also be adsorbed by the silica gel. The silica gel releases (desorbs) the adsorbed water vapor upon heating to 177°C and can be reused.

Often the condenser and drying tube are used in series to increase water vapor collection efficiency and obtain a high capacity for the water removed. Large mesh silica gel (6-16) is used with a filter support backing to prevent the possibility of entrainment of small particles which might damage other components.

## Heating

The heating operation is used solely to preserve the gas sample prior to passing it through the particulate collector. This is an effective method of preventing condensation of water vapor and high molecular weight substances. Therefore it is common practice to heat sampling probes and particulate collectors such as filters to prevent deposition by condensation. If such a condensation process were allowed to occur, it would cause loss of gaseous constituents from the gas sample. In addition to causing a sampling error these materials could be deposited in accessible areas of the sampling system and lead to later malfunctions.

Ideally it is a good policy to try to maintain stack gas temperatures throughout the sampling train preceding the filter. However, high temperatures favor chemical reactions such as oxidation of hydrocarbons in a gas stream containing appreciable amounts of oxygen. Low temperatures, as mentioned, are conducive to condensation of water vapor and high molecular weight hydrocarbons. Hence a compromise is required and most probes and heated filter boxes operate at about 121°C. Heat sensitive sampling train components will not be affected by this temperature. Water vapor will not condense and some of the safety problems involved with the handling of hot equipment will be alleviated. Particulate compliance tests require that a gas stream temperature no higher than 115°C be maintained prior to particulate filtration. Any condensables which are taken out by the probe and filter under this condition are considered to be part of the particulate catch. A recent ruling however, allows the use of temperature up to 120°C when testing at fossil fuel utility boilers.

## Dilution

Addition of a dry gas can be an effective method for preventing condensation. When this gas is added the sample is diluted. Condensation is prevented because the dry gas is capable of supporting a part of the water vapor from the gas sample even though the temperature of the mixture is reduced. The dry gas must be added in such a manner that the original sample constituents are not altered. This could be a problem with respect to particulate matter because the dilution (mixing) process could cause such events as particle agglomeration, deposition, and condensation.

## 8.1.11 Pumps

The purpose of a pump is to pull the sampled gas through the sampling train components. The detail of the particular type of pump required will depend on several criteria. The pump must provide adequate flow and pressure characteristics and be durable and portable. The pump must be able to overcome the pressure drop of the other sampling train components and thereby provide the desired flowrate. It must be able to provide a wide range of flowrates as required by isokinetic sampling conditions. Often the head loss across the filter increases through the sampling tests. This puts an added burden on the pump which must still be able to maintain the required sampling rate at the nozzle tip within the stack.

The pump must be leakless when it is located ahead of the gas meter in the sampling train. If it isn't, then the metered volume will be greater than the sampled volume and hence the measured particulate concentration will be less than the true particulate concentration. The EPA Method 5 sampling train falls in this category. In many of the other sampling trains, the pump is located after the gas meters and therefore no error is involved if a leak exists in the pump.

The pump must be durable in that it is exposed to corrosive environment of the sample gas. During most source tests it is in constant operation and should be a long life component. The design should enable this component to be maintained easily; the key components should be accessible and replaceable with a minimum amount of time.

The need for portability becomes readily apparent when performing source tests, and consequently a small, lightweight pump is desirable.

There are several types of pumps suitable for source sampling trains. All are of positive displacement types which are capable of producing relatively high vacuums (>686 mm of Hg below atmosphere pressure) and operate with a direct linear correspondence between the flow rate and inlet pressure. In commercial source sampling equipment, reciprocating diaphragm and rotary vane pumps are commonly used.

The diaphragm pump operates on the moving diaphragm principle. Gas is drawn into the chamber on a suction stroke and pushed out on the discharge stroke. On the suction stroke a suction valve is open, allowing gas to flow in. On the discharge stroke the suction valve closes and a discharge valve opens allowing the gas to flow out. This intermittent operation can cause some flow fluctuation (pulsation) in the sampling train. However, this problem can be somewhat reduced by running two such pumps in parallel or by a specifically designed surge chamber in the flow line. The diaphragm in these pumps is made out of metal, rubber or plastic.

The rotary vane pump is one rotor in a casing, which is machined eccentrically in relation to the shaft. The rotor contains a series of movable vanes which seal against the pump casing. The vanes are free to slide in and out of the slots as the rotor turns. If the pump must be leakless, then only the fiber vane type pump with an oiler should be used. The oiler may have to be modified so that no ambient air leaks into the system through the oil bowl.

### 8.1.12 Flow Control

Flow regulation for most sampling trains is accomplished by using a throttling valve preceding the pump. This valve varies the vacuum the pump must work against and thereby changes the flow rate. A more sophisticated arrangement uses two valves. One precedes the pump and provides a coarse control while a second one is installed in a recycle (by-pass) loop to protect the pump and provide a fine control. This latter arrangement is used in the EPA Method 5 particulate sampling train. This double valve arrangement is also easier on the pump, allowing longer pump life.

The major requirements of the flow control valve are: (a) it allows sensitive flow rate adjustment to meet proportional sampling (isokinetic) conditions and (b) it does not allow any leakage. Both these requirements depend upon the valve construction. The leakage problem poses the same potential error as was discussed for the pumps. Good quality needle valves are required in most source sampling applications.

## 8.2 ELECTROSTATIC PRECIPITATOR SAMPLING-PRELIMINARY PROCEDURES

### 8.2.1 Introduction

Before a sample is taken, several preliminary tests must be made to determine some of the characteristics of the sampling location and the gas stream. The results of these preliminary tests are the basis for determining suitability of the sampling location, the nozzle size, number of sampling points, sampling time, and nomograph settings.

### B.2.2 Physical Sampling Location Characteristics

The sampling location should be at a position where the gas flow is sufficiently uniform that an accurate sample can be obtained. Eight to ten duct diameters downstream and two duct diameters upstream from any disturbance such as bends, duct inlets, duct outlets, or changes in diameter should give sufficiently uniform flow. If the flow at the sampling location is very uniform, a minimum number of twelve sample points per sample may be used. If the upstream and downstream diameter requirement is not met, the flow at the sampling location is likely to be very non-uniform and require an increase in the number of sampling points. In such a case, Figure B7 is used as a guide to determine the number of sample points necessary to define the velocity profile adequately. Figure B7 is used by reading the number of sampling points corresponding to both the number of downstream (A) and upstream (B) diameters and selecting the greater number of sampling points. A quick pitot tube survey should indicate whether a sufficient number of points has been chosen to define the velocity profile adequately. After the number of sample points is selected, the cross section of the duct is divided into a number of equal areas as shown in Figure B8. In the case of round stacks, the sample point is located such that half of the area increment represented by that point is radially on each side of the sample point. The location of sampling points is determined as shown in Table B1. The area increments must be small enough to insure that the flow at the sampling point in each area is representative of the flow in the area; however, the total number of area increments must be limited enough so that all the points may be sampled within a reasonable period of time.

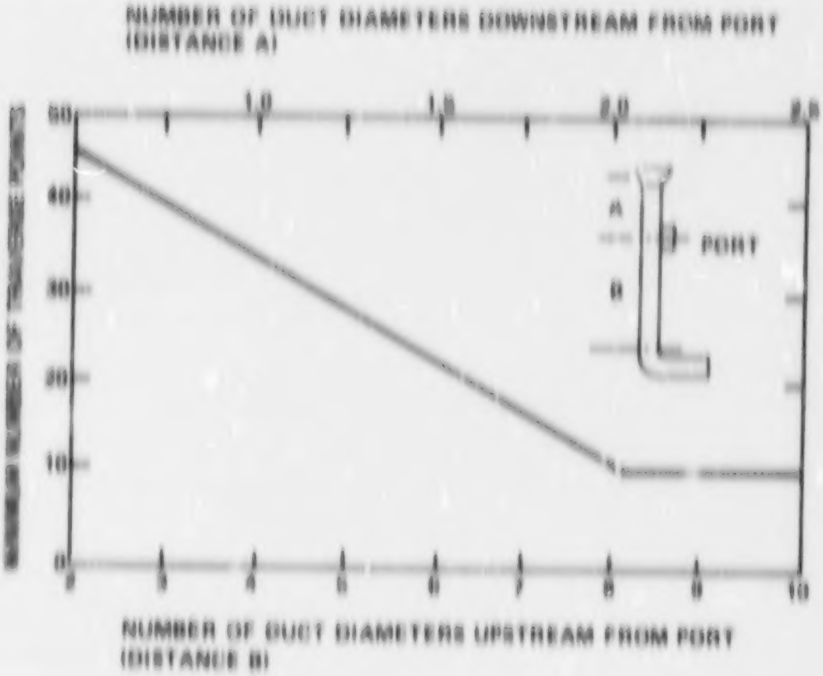
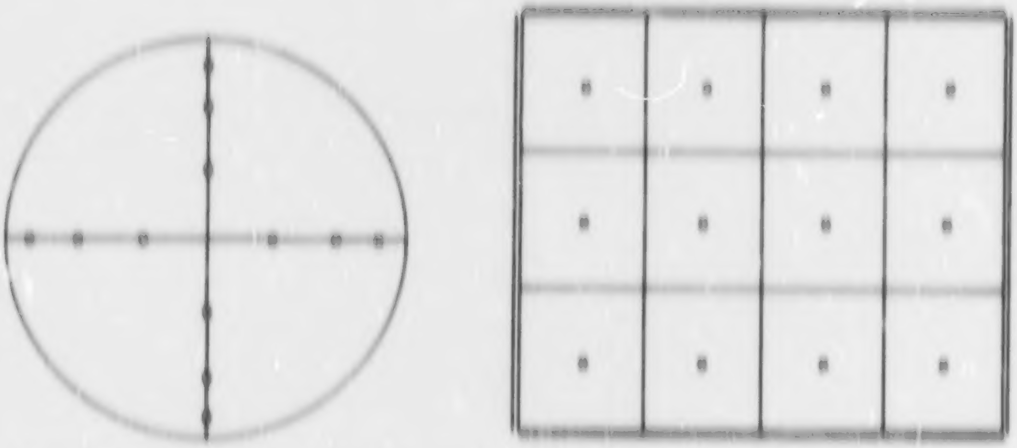


*Figure B7.* *Minimum number of traverse points per sample obtained from "Distances to Disturbances", upstream and downstream.*



CROSS SECTION OF CIRCULAR DUCT DIVIDED INTO 12 EQUAL AREAS, SHOWING LOCATION OF TRAVERSE POINTS.

CROSS SECTION OF RECTANGULAR DUCT DIVIDED INTO 12 EQUAL AREAS, WITH TRAVERSE POINTS AT CENTROID OF EACH AREA.

*Figure 88. Examples of equal area sample points.*

# TABLE B1

## Duct Traversing Length Factors

LENGTH FACTORS, $R_L$

(Fraction of stk. diam. from inside wall to traverse pt.)

| Traverse Point Number | NUMBER OF TRAVERSE POINTS ON A DIAMETER | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 2 | 4 | 6 | 8 | 10 | 12 | 14 | 16 | 18 | 20 | 22 | 24 |
| 1 | .146 | .067 | .044 | .033 | .025 | .021 | .018 | .016 | .014 | .013 | .011 | .011 |
| 2 | .854 | .250 | .147 | .105 | .082 | .067 | .057 | .049 | .044 | .039 | .035 | .032 |
| 3 | | .750 | .295 | .194 | .146 | .118 | .099 | .085 | .075 | .067 | .060 | .055 |
| 4 | | .933 | .705 | .323 | .226 | .177 | .146 | .125 | .109 | .097 | .087 | .079 |
| 5 | | | .853 | .677 | .342 | .250 | .201 | .169 | .146 | .129 | .116 | .105 |
| 6 | | | .956 | .806 | .658 | .355 | .269 | .220 | .188 | .165 | .146 | .132 |
| 7 | | | | .895 | .774 | .645 | .366 | .283 | .236 | .204 | .180 | .161 |
| 8 | | | | .967 | .854 | .750 | .634 | .375 | .296 | .250 | .218 | .194 |
| 9 | | | | | .918 | .823 | .731 | .625 | .382 | .306 | .261 | .230 |
| 10 | | | | | .975 | .882 | .799 | .717 | .618 | .388 | .315 | .272 |
| 11 | | | | | | .933 | .854 | .780 | .704 | .612 | .393 | .323 |
| 12 | | | | | | .979 | .901 | .831 | .764 | .694 | .607 | .398 |
| 13 | | | | | | | .943 | .875 | .812 | .750 | .685 | .602 |
| 14 | | | | | | | .982 | .915 | .854 | .796 | .739 | .677 |
| 15 | | | | | | | | .951 | .891 | .835 | .782 | .728 |
| 16 | | | | | | | | .984 | .925 | .871 | .820 | .770 |
| 17 | | | | | | | | | .956 | .903 | .854 | .806 |
| 18 | | | | | | | | | .986 | .933 | .884 | .839 |
| 19 | | | | | | | | | | .961 | .913 | .868 |
| 20 | | | | | | | | | | .987 | .940 | .895 |
| 21 | | | | | | | | | | | .965 | .921 |
| 22 | | | | | | | | | | | .989 | .945 |
| 23 | | | | | | | | | | | | .968 |
| 24 | | | | | | | | | | | | .989 |

In rectangular ducts or stacks, the cross section is divided into a number of equal area rectangles. The sample is taken at the centroid of each rectangular area. These areas should be laid off such that the ratio of the length to the width of the elemental areas is between one and two.

The minimum number of sample points is twelve, and the same criteria as for round ducts is used to insure that the velocity profile is adequately defined. The equivalent diameter of a square duct is approximated by 2LW/L+W where L and W are the duct cross-sectional dimensions.

In most cases the dimensions of the ducts obtained from construction drawings are accurate; however, the inside dimensions should still be measured if feasible, particularly in the case of horizontal ducts on the bottoms of which dust deposits of considerable thickness are often found. The pitot tube may be used to make this measurement but the ends should be protected to prevent material from the back wall from clogging the ends. Another critical measurement is the length of the port extension. With these measurements, the pitot tube is marked at the points to be sampled. If the outermost points are less than one inch from the walls, they should be located at one inch and noted on the sampling form. The required length of the probe (pitot tube) for each of the points may be marked with hose clamps, tape, or other suitable material compatible with flue gas conditions.

Stack pressure is determined with a leveled and zeroed manometer. The pitot tubes are aligned perpendicular to the flow stream in the stack and one of the two pitot lines is disconnected from the console. If the stack pressure is positive gauge pressure, the manometer will show positive deflection with the one pitot line connected to the positive side of the manometer. If the stack pressure is negative gauge pressure, the manometer will show positive deflection with the one pitot tube line connected to the negative side of the manometer. The stack gauge pressure ($P_S$) in millimeters of Hg is obtained by adding the stack to ambient differential pressure, $\Delta P_D$, to the ambient pressure ($P_{AMB}$).

$$P_S = \Delta P_D + P_{AMB}$$

The preliminary flue gas temperature is obtained by a suitable means such as a stem thermometer placed in the sampling port or a thermocouple with an appropriate readout device.

### B.2.3 Velocity Determination

Before velocity measurements are taken, the inclined manometer must be leveled and zeroed and must remain level during sampling. The openings of the pitot tubes should be shielded from any wind currents but not be completely closed off when the manometer is zeroed. Correct connection of the pitot tube lines may be checked by blowing gently on the upstream pitot tube opening and noting the response on the manometer. The probe is then inserted, the pitot reading noted and the pitot tube lines are switched both on the console and on the probe. If the manometer reading is the same as that prior to switching the pitot tube lines it is reasonably certain that there is no significant leak in the lines. If a leak is detected, it must be eliminated before any readings are taken.

The pitot tube lines must not be pinched or the tube stopped up during the traverse. If fluctuations in the manometer are noted, pieces of cotton or glass wool may be placed in the pitot tube lines to dampen the fluctuation but should not be packed too tightly. Since the pitot tube measures pressure differences, there is no actual air flow through the lines. If one line is completely plugged, however, the results will not be accurate. In some instances condensation of water within the pitot tube lines can cause difficulties and erroneous readings.

Once the traverse has been completed and the pressure and temperature readings have been recorded the velocity may be determined. Velocity may be calculated as follows:

$$\left(V_s\right)_{avg} = (422.67)(C_p)\left(\frac{(T_s)_{avg}(\Delta P)_{avg}}{P_s M_s}\right)^{\frac{1}{2}}$$

where $(V_s)_{avg}$ is the stack gas velocity, cm/sec,

$T_s$ is the average stack temperature, °K,

$\Delta P$ is average stack gas velocity head, mm Hg,

$M_s$ is the molecular weight of the stack gas, wet basis, gm/gm-mole,

$P_s$ is the absolute stack pressure, mm Hg, and

$C_p$ is the pitot tube correction factor.

### 8.2.4 Stack Moisture Content

The stack moisture content is an important factor in stack sampling. Nozzle size selection and sampling rate are both dependent on the moisture content. A condenser method or a determination (based on the dry bulb temperature with knowledge that saturated conditions exist in the stack) are two ways of determining moisture content. A wet bulb-dry bulb technique requires less equipment but must be limited to non-acid gas streams with moisture contents of less than 15% and dew points less than 52°C. The condenser method works well for most gas streams and is relatively easy to perform.

### B.2.4.1 Condenser Method

Several condenser techniques can be used to determine stack moisture content. One such technique uses a Greenburg-Smith impinger approximately half full of water followed by a straight impinger approximately half full of silica gel. A measured volume of stack gas, usually 10 cubic feet, is drawn through the impingers at a moderate flow rate. The total change in weight of the impingers is the weight of the moisture caught. The impingers should be in an ice bath while the stack gas is drawn.

With the impinger volume increase in milliliters, $V_{wo}$, the stack gas volume in cm³ ($V_m$) corrected by the dry gas meter correction factor, the absolute average meter temperature in °K ($T_m$), and the meter pressure in mm of mercury ($P_m$), the moisture fraction ($B_{wo}$) is calculated as follows:

$$B_{wo} = \frac{V_{wo}\ (1243.34\ \mathrm{cm^3/ml(H_2O)})}{V_{wo}\ (1243.34\ \mathrm{cm^3/ml(H_2O)}) + V_m\left(\frac{294.4}{T_m}\right)\left(\frac{P_m}{760\ \mathrm{mm}}\right)}$$

### B.2.4.2 Saturation Method

If water droplets are present in the stack and the stack gas temperature is below 100°C, the gas stream may be assumed to be saturated. The moisture content is read from the saturation curve on the psychrometric chart at the stack gas temperature, Figure B9.

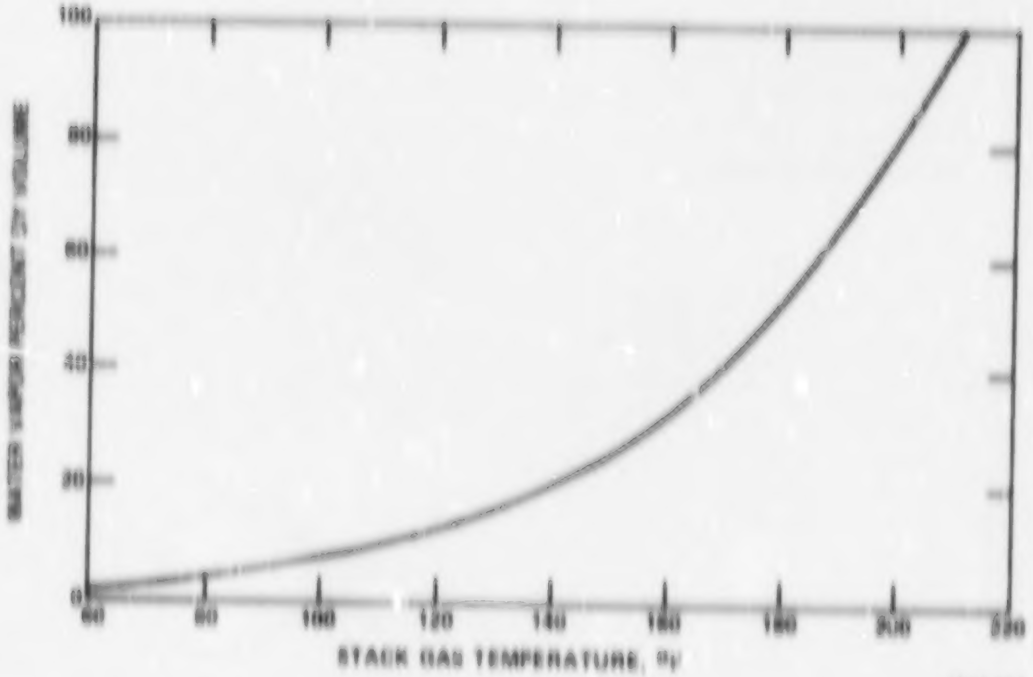


*Figure B9. Percent water vapor in air at saturation.*

### B.2.4.3 Wet Bulb-Dry Bulb Method

In this method two thermometers are placed in the gas stream. One is dry and the other has a wet sock over the bulb. The temperatures are read after they stabilize. If the stack or duct pressure is near atmospheric pressure, the percent moisture may then be found from the psychrometric chart, Figure B10.

The percent water vapor by volume is found directly on the ordinate axis. Inputs are the dry bulb temperature on the abscissa, the wet bulb temperature, and the sloping lines which terminate at the saturated vapor line.

When obtaining wet bulb-dry bulb readings with a sling psychrometer, the plane of the thermometers should be perpendicular to the flow of gas. If it is parallel to the flow, the dry bulb should be upstream of the wet bulb. The gas velocity past the wet bulb should be from 3.7 to 9.3 meters per second. Sufficient time must be allowed for the wet bulb temperature to stabilize or inaccurate results will be obtained.

### B.2.5 Molecular Weight of the Stack Gas

The most common method of determining the composition of combustion effluents is the Orsat apparatus. Although flue gases vary in composition, they normally contain $CO_2$, CO, $O_2$, $H_2O$, and $N_2$. The Orsat analysis determines the quantities of these components (except $H_2O$) present by successive removal using suitable absorbents and measurement of the volume changes of the original sample. The Orsat analysis, as it is normally used, measures the percentage of $CO_2$, $O_2$, and CO in the sample. The difference is largely $N_2$. By changing the absorbents other components may also be measured.

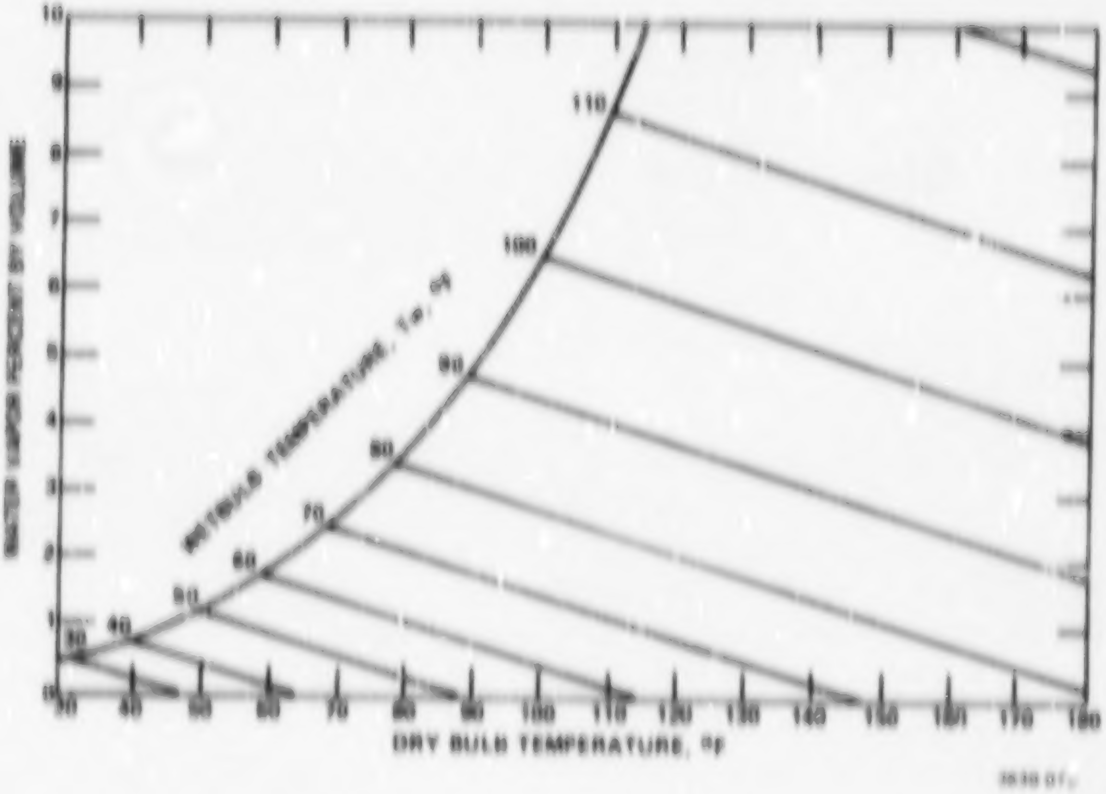


*Figure B10. Percent water vapor with wet and dry bulb.*

After the Orsat analysis has been performed, the molecular weight of the stack gas may be determined by the following equation:

$$M_S = (B_{WO})(18.0) + (B_{DG})\,\Sigma \begin{bmatrix} \text{proportion by vol.} \\ \text{of component on} \\ \text{dry gas basis} \end{bmatrix} \times \begin{bmatrix} M_{SC}\text{ of} \\ \text{component} \end{bmatrix}$$

where $M_S$ is the molecular weight of the stack gas,
$B_{DG}$ is dry gas fraction of the stack gas, and
$M_{SC}$ is the molecular weight of each component of the stack gas.

## B.3 ELECTROSTATIC PRECIPITATOR SAMPLING FOR PARTICULATE AND GASES

### B.3.1 *Isokinetic Sampling*

To obtain a representative particulate sample, the sample must be collected at a rate as nearly isokinetic as possible, i.e., the kinetic energy of the gas stream in the stack is equal to the kinetic energy of the gas stream through the sampling nozzle. Since the composition of the two gas streams is the same, this energy balance simplifies to: the velocity in the stack is equal to the velocity through the nozzle. If a particulate sample is not pulled isokinetically, inaccurate results may be obtained.

Whenever an object is placed in a moving gas stream, some disturbance of the flow patterns will occur. The purpose of isokinetic sampling is to minimize any disturbance caused by the sampling nozzle. A sample collected isokinetically through a sharp-edged nozzle, should create very little disturbance. Figure B11 illustrates this point.

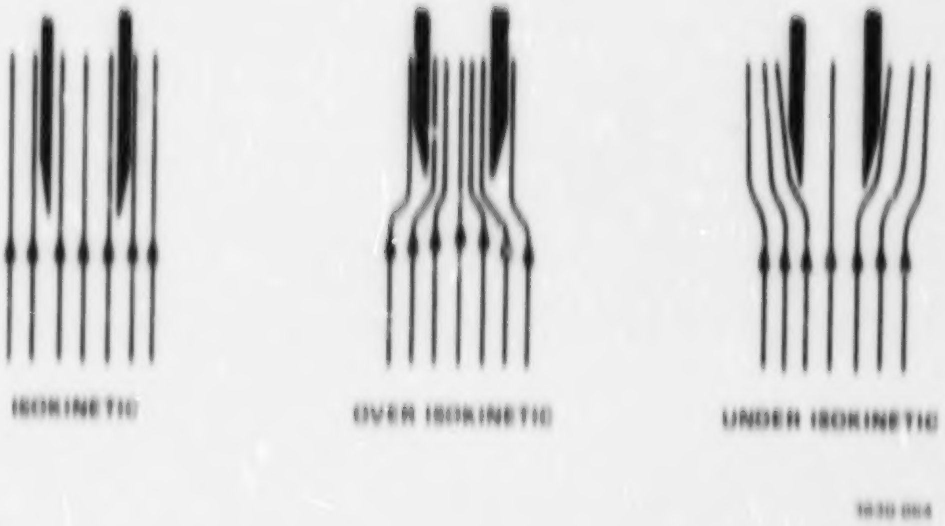

ISOKINETIC
OVER ISOKINETIC
UNDER ISOKINETIC

*Figure B11. Isokinetic flow patterns.*

Large (heavy) particles tend to travel in a straight line and are not greatly affected by flow disturbances, whereas small (light) particles tend to follow the flow lines. In a gas stream with a homogeneous distribution of large and small particles, over-isokinetic sampling will give a low particulate mass rate (PMR) because fewer large particles will be caught than are representative of the flow stream from which the gas was withdrawn. On the other hand, under-isokinetic sampling will give a high PMR due to a greater than representative number of large particles that will be caught.

The velocity of a gas stream in a stack generally varies from point to point; therefore, the flow rate or velocity through the sampling nozzle must be adjusted to maintain isokinetic conditions at each sampling point. In the sampling train, determination of the nozzle volume and the flow rate through the nozzle are based on dry gas volume and flow rate measured at approximately ambient temperatures. For this reason, the flow rate through the orifice meter which corresponds to the desired flow rate through the sampling nozzle must be determined. The stack velocity as measured by the pressure drop (ΔP) across the pitot tube and the velocity through the nozzle as measured by the pressure drop (ΔH) across an orifice meter at the end of the sampling train must be equal in order to maintain isokinetic flow. To speed up this calculation, two nomographs were developed by the old National Air Pollution Control Administration (NAPCA). Through the use of these nomographs, the proper size sampling nozzle may be selected and the flow adjustments required to maintain isokinetic sampling conditions may be determined. An alternate method employs the use of one of the NAPCA nomographs or a calculator for adjusting flow rates along with a mathematical method of nozzle selection. These two procedures give equivalent results.

### 8.3.2 Sampling For Effluent Gases

When effluent gases and particulates are to be sampled simultaneously the sampling must be performed isokinetically. It is advisable however, to choose a nozzle which will give a low volumetric flow rate to optimize gas absorption efficiency in the impinger train; 142 cm³/sec (.3 ft³/min) or less is desirable. If only gases are to be sampled and if they are well mixed, isokinetic sampling is not necessary.

### 8.3.3 Nozzle Selection and Nomograph Setting

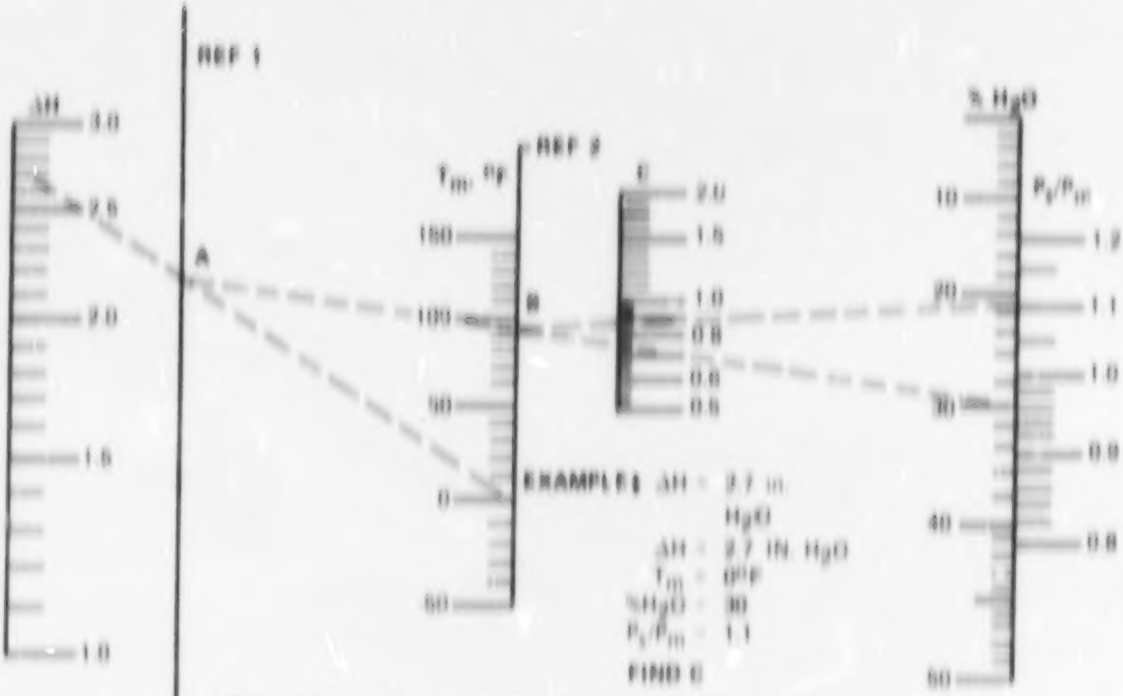
Isokinetic sampling involves maintaining the flow rate through the sampling nozzle such that the velocity in the nozzle equals the velocity in the stack at the sampling point. Obviously, the flow rate through almost any size nozzle could be adjusted such that this velocity requirement is met. There are certain physical limitations, however, placed on nozzle size by the sampling equipment: pump capacity, port diameter, filter efficiency, and the critical flow through the Greenburg-Smith impingers. Another limiting factor involves the reliability of the sample. Small nozzles can yield less representative samples when large particles are present. As the ratio of nozzle tip area to stack cross-sectional area decreases, the chance of sampling at a point where the flow is not representative of the flow in the stack area that the point represents increases. The nozzle should also be at least a little larger than the largest particles that might be encountered in the stack. Some guidelines for nozzle size selection are given in the next section.

### B.3.4 EPA Reference Method 5 Procedure

Isokinetic sampling, the condition of equal velocities, implies a mathematical relationship between the two pressure drops, ΔP and ΔH. The pressure drop measured by the pitot tube, ΔP, indicates the stack velocity and the desired velocity through the sampling nozzle. The pressure drop across the orifice plate, ΔH, represents the flow rate of dry gas through the dry gas meter. This relationship has been incorporated into two stack sampling nomographs, Figures B12 and B13. To operate the nomographs, a factor which is a composite of the test constants is obtained from one nomograph and is used to set up the second nomograph. This factor will be called "C". As the pressure drop across the pitot tube (ΔP) changes from point to point, an updated desired value for the orifice pressure drop (ΔH) is found from the second nomograph. The flow rate may then be adjusted to give this desired pressure drop across the orifice which establishes isokinetic conditions.

In using the first nomograph, the following parameters are required to determine the "C" factor which will be carried over to the second nomograph:

1. Pressure drop (ΔH) across the orifice plate when 0.75 cubic feet per minute of dry gas is flowing and the pressure and temperature are 760 mm mercury and 21.1°C respectively is determined. Note that the calibration factor of the orifice must be determined in the lab.


REF 1
ΔH
3.0
2.5
2.0
1.5
1.0
A
REF 2
$T_m$, °F
150
100
50
0
-50
B
C
2.0
1.5
1.0
0.8
0.6
0.5
% $H_2O$
10
20
30
40
50
$P_s/P_m$
1.2
1.1
1.0
0.9
0.8

EXAMPLE: ΔH = 2.7 in. $H_2O$

ΔH = 2.7 IN. $H_2O$
$T_m$ = 0°F
%$H_2O$ = 30
$P_s/P_m$ = 1.1
FIND C

DRAW LINE FROM ΔH TO $T_m$(°F) TO OBTAIN POINT A ON REF. 1.
DRAW LINE FROM POINT A TO %$H_2O$ AND READ B ON REF. 2.
DRAW LINE FROM POINT B TO $P_s/P_m$ AND OBTAIN ANSWER OF 0.85 FOR C

3830 081

*Figure B12. Stack Sampling Nomograph (side 1)*

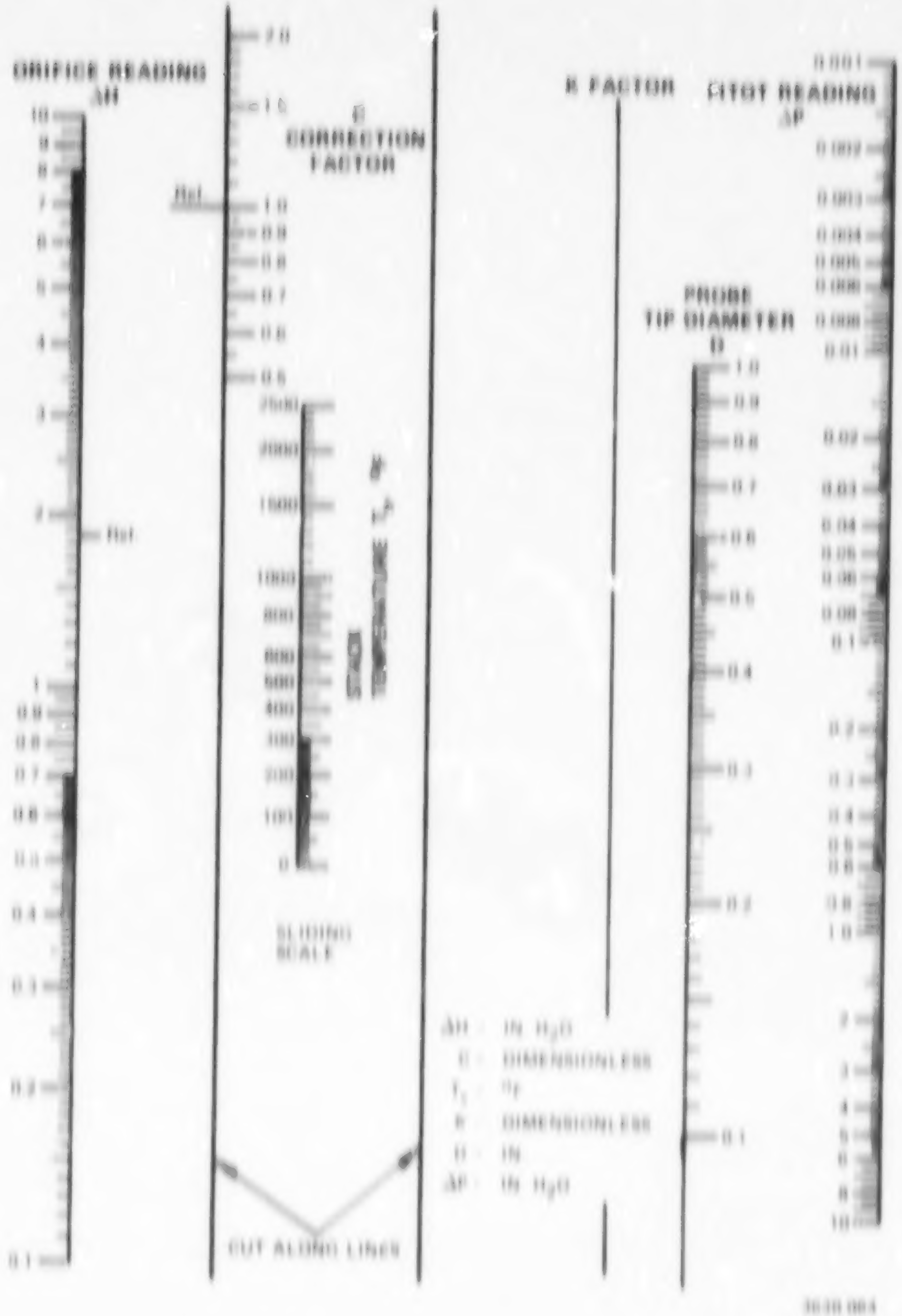

ORIFICE READING ΔH
C CORRECTION FACTOR
Ref
Ref
STACK TEMPERATURE $T_s$, °F
SLIDING SCALE
CUT ALONG LINES
K FACTOR
PITOT READING ΔP
PROBE TIP DIAMETER D
ΔH = IN $H_2O$
C = DIMENSIONLESS
$T_s$ = °F
K = DIMENSIONLESS
D = IN
ΔP = IN $H_2O$
3630 004

*Figure B13 Stack Sampling Nomograph (side 2)*

2. Percent $H_2O$ in the stack gas is determined prior to sampling.

3. Expected temperature at the orifice must be estimated from experience. It is usually at least ten degrees Fahrenheit higher than ambient temperature and is the same as the outlet temperature from the dry gas meter.

4. The ratio of absolute stack pressure to atmospheric pressure is also required. This ratio is approximately equal to one.

Proceed to find "C" as follows:

A. Obtain a point on the "reference one" line by connecting the orifice pressure drop, ΔH, (for 0.75 cubic feet per minute) to the temperature of the orifice plate ($T_m$).

B. Draw a line from this point on the "reference one" line to the percent $H_2O$ to obtain a point on the "reference two" line.

C. Connect this point to the pressure ratio point; this line crosses the "C" scale and gives the value required to set up the second nomograph.

After the initial velocity traverse, and prior to sampling, set up the second nomograph as follows:

A. Set the vertical sliding scale so that the "C" factor mentioned above is at the reference point.

B. From the velocity traverse, calculate the average ΔP. A line from this point to the stack temperature intersects the probe tip (nozzle) diameter scale.

C. From the nozzles on hand, select one near this size. Reconstruct the line through the stack temperature and use the selected probe tip diameter to give a reference point on the ΔP scale.

D. A line from the reference point on the ΔP scale to the permanent reference point on the ΔH scale locates the pivot point on the K factor line. Lock the pivot bolt that carries the clean plastic rule at this point. The nomograph is now set up for isokinetic sampling. Note that stack temperature is assumed to remain constant.

For each point of the sampling traverse the pressure drop across the pitot tube, ΔP, is used as input to the nomograph. The output is the desired pressure drop across the orifice, ΔH, which is required to maintain isokinetic flow. If the temperature of the stack changes appreciably during sampling, (±5% on an absolute temperature basis) the pivot point must be reset. Once the pivot point on the K factor line has been set, it is suggested that the maximum and minimum ΔP's from the velocity traverse be used to determine the range of ΔH's. This range should lie between 0.3 and 6 inches of $H_2O$. If it does not, a different choice of nozzle tip diameter should be considered.

### 8.3.5 General Sampling Procedures

#### 8.3.5.1 Preparing the Glassware

The glassware must be prepared and placed in the sampling box. All of the glassware should be clean and dry. The glass liner in the probe should be washed and the nozzle attached to the probe. In the sampling box, the sample first passes through a glass cyclone which has a flask attached to the bottom to catch large particles that the cyclone separates from the gas stream. The sample then goes through a fritted glass filter holder. The filter holder is 6.4 cm to 10 cm in diameter. A glass fiber filter is placed over the fritted surface. The filter should be preweighed to ±0.0001 grams. From the filter the sample goes into the condenser section. The type and number of impingers to be used in this section is dictated by the type of sample to be taken. The different impinger solutions and their uses are listed in the following paragraphs. Gummed labels can be used to label the impingers and should be placed near the top to prevent water from being absorbed from the ice slurry. The impingers should be weighed to ±0.1 gram before and after sampling. A very light coating of silicone lubricant should also be placed on the ball joints connecting the glassware to insure a vacuum-tight seal. The impinger solution volume is normally 200 ml.

### 8.3.5.2 Impinger Trains

The following tables give some recommended impinger (IMP) trains.

Preparation of Impinger Solutions

80% Isopropanol: 160 ml Technical Grade isopropanol + 40 ml deionized (DI) water.
6% $H_2O_2$: 40 ml Reagent Grade 30% Hydrogen Peroxide + 160 ml DI water.

0.1 N NaOH: 20 ml 1 N NaOH + 180 ml DI water.

1 N NaOH: 40 grams Reagent Grade NaOH (pellet form) dissolved in DI water made up to 1.0 liter volume.

Alkaline arsenite solution (0.500 N $NaAsO_2$ in 2.5 N NaOH): Dissolve 100 grams Reagent Grade NaOH (pellet form) and 32.5 grams of $NaAsO_2$ in DI water and dilute to 1.000 liter with DI water.

0.1 N $H_2SO_4$: 10 ml 2 N $H_2SO_4$ diluted with DI water to 200 ml. Prepare 2 N $H_2SO_4$ by adding 1 volume of Reagent Grade concentrated $H_2SO_4$ (18 M) to 17 volumes of DI water.

0.1 M zinc acetate: 22 grams $Zn(C_2H_3O_2)_2 \cdot 2H_2O$ in 1.0 liter DI water.

I. Particulate Only

Application: Hot mix plants, ore sintering processes, gypsum manufacturing, etc.

| IMP. NO. | CONTENTS | POLLUTANT OR COMPONENT | IMPINGER TIP CONFIGURATION |
|---|---|---|---|
| 1 | DI Water | Particulate | Straight |
| 2 | DI Water | Particulate | Greenburg-Smith (G-S) |
| 3 | Dry | Water | Straight |
| 4 | Silica Gel | Water | Straight |

II. Particulate + Sulfur Dioxide

Application: Non-ferrous smelters, Portland cement kilns, coal-fired boilers, fluid catalytic cracking units, sulfuric acid plants, boiler recovery stacks (kraft paper mills).

| IMP NO. | CONTENTS | POLLUTANT OR COMPONENT | IMPINGER TIP CONFIGURATION |
|---|---|---|---|
| 1 | 80% isopropanol | Particulate, $H_2SO_4$, $SO_3$ | Straight |
| 2 | 6% $H_2O_2$ | $SO_2$ | G-S |
| 3 | 6% $H_2O_2$ | $SO_2$ | G-S |
| 4 | Dry | Water | Straight |
| 5 | Silica Gel | Water | Straight |

NOTES: 1. The impinger train must be purged with two cubic feet of ambient air at the end of the run to sweep $SO_2$ out of impinger #1 into impingers 2 and 3.

2. The first impinger not only is effective in trapping sulfuric acid but will also trap submicron particulate that passes through the filter.

III. Particulate + Chlorine and/or Chlorides

Application: Pulp bleaching (effluent from), Magnesium plants (drying of magnesium chloride).

| IMP NO. | CONTENTS | POLLUTANT OR COMPONENT | IMPINGER TIP CONFIGURATION |
|---|---|---|---|
| 1 | DI Water | HCl, $Cl^-$<br>Particulate | G-S |
| 2* | Alkaline Arsenite Solution | HCl, $Cl_2$<br>Particulate | G-S |
| 3 | Alkaline Arsenite Solution | HCl, $Cl_2$ | G-S |
| 4 | Dry | Water | Straight |
| 5 | Silica Gel | Water | Straight |

*NOTE: It is important to measure the volume of this solution as accurately as possible since the analysis will be based on the molar quantity of arsenite remaining.

## IV. Particulate in the Presence of Hydrogen Fluoride

Application: Alumina reduction plants and phosphate fertilizer manufacturing (acidulation process).

| IMP NO. | CONTENTS | POLLUTANT OR COMPONENT | IMPINGER TIP CONFIGURATION |
|---|---|---|---|
| 1 | Water | Particulates, inorganic fluoride particulate | G-S |
| 2 | Water | Particulates, inorganic fluoride particulate | G-S |
| 3 | Dry | Water | Straight |
| 4 | Silica Gel | Water | Straight |

NOTE: Use filter bypass instead of fritted filter.

## V. Particulate + Ammonia

Application: Effluent from manufacturing, prilling and drying of ammonium nitrate or ammonium phosphate fertilizer.

| IMP NO. | CONTENTS | POLLUTANT OR COMPONENT | IMPINGER TIP CONFIGURATION |
|---|---|---|---|
| 1 | Water | Particulate | G-S |
| 2 | 0.1 N $H_2SO_4$ | $NH_3$ | Straight |
| 3 | 0.1 N $H_2SO_4$ | $NH_3$ | Straight |
| 4 | Dry | Water | Straight |
| 5 | Silica Gel | Water | Straight |

NOTE: Water is used in the first impinger in order that particulate may be measured. The contents of this impinger will be saturated with ammonia. Partial recovery of ammonia from the first impinger may be accomplished by purging the impinger train with approximately 2 ft$^3$ of ambient air.

VI. Particulate + Hydrogen Sulfide

A. Application: Carbon black plants (furnace effluent), lime kilns (kraft paper mills).

| IMP NO. | CONTENTS | POLLUTANT OR COMPONENT | IMPINGER TIP CONFIGURATION |
|---|---|---|---|
| 1 | 0.1 M zinc acetate | $H_2S$ | G-S |
| 2 | 0.1 M zinc acetate | $H_2S$ | G-S |
| 3 | Dry | Water | Straight |
| 4 | Silica Gel | Water | Straight |

B. Application: kraft pulp mills, boiler recovery stacks.

| IMP NO. | CONTENTS | POLLUTANT OR COMPONENT | IMPINGER TIP CONFIGURATION |
|---|---|---|---|
| 1 | Water | Particulate<br>$SO_3$, $H_2SO_4$ | Straight |
| 2 | 0.1 M zinc acetate | $H_2S$ | G-S |
| 3 | 0.1 M zinc acetate | $H_2S$ | G-S |
| 4 | Dry | Water | Straight |
| 5 | Silica Gel | Water | Straight |

NOTE: With a conventional sampling train $H_2S$ and $SO_2$ cannot be determined simultaneously.

### 8.1.5.3 Checking the Sampling Train for Leaks (Vacuum Check)

A vacuum-tight system is necessary to prevent any dilution air from being pulled into the sampling line. After the probe and sample box are connected and suspended from the monorail or other support, the umbilical cord containing the sample line, pitot tube lines and thermocouple leads may be connected. The inlet side of the cyclone is then sealed for the vacuum check. The pump is then started with the coarse valve closed and bypass valve open. As the coarse valve is slowly opened the vacuum will begin to increase. The bypass valve is slowly closed until the vacuum reaches 381 mm gauge. At this vacuum the flowrate through the dry gas meter should not exceed 9 $cm^3$/sec (.02 $ft^3$/min). If a leak is present all connections should be checked to eliminate the leak and the above procedure repeated. The seal on the cyclone must be removed slowly before the pump is turned off to prevent liquid backup in the impingers. After the above vacuum check is performed the probe liner is connected to the cyclone and a vacuum check on the total system may be performed. The pump is started again and the coarse valve is slowly opened. After the flow starts, the nozzle tip is sealed and vacuum should start to build in the system. When the sample line vacuum reaches 381 mm the coarse valve should be closed. The vacuum should hold steady if there are no leaks. If it holds for about fifteen seconds, the nozzle tip is opened and the vacuum should drop. Then the pump is turned off with care not to back up liquid in the impingers. Under no circumstances should the pump be turned off while the nozzle tip is sealed. The power line is then plugged in and the heater turned on. The probe heater, if used, should be connected and the probe heater turned on. The pitot tube manometer may then be zeroed by shielding the end of the pitot tube from any wind or disturbance. The ends should not be plugged, however. The orifice manometer should also be zeroed. The manometer must be level at this time and throughout the sampling period. The pitot tube should also be checked to be sure the lines are hooked up properly. The man on the platform can blow lightly on the upstream pitot tube while the console operator checks the manometer displacement. Before sampling begins, the console operator should be sure the manometers are leveled and zeroed, the temperature indicator is working properly, and the nomograph or calculator is properly set up.

The console operator should have decided on how long to sample at each point. The number of sample points is determined by generally accepted rules, e.g. velocity traverse, but the sampling time is based on knowledge of the plant operations and the approximate particulate loading which may be obtained from plant personnel on a pretest. The sample collected on the filter should be large enough to weigh accurately and be representative of the conditions in the stack; however, care must be exercised to prevent the filter from clogging, or in wet stacks, the impingers from filling up. It is preferable to sample for at least an hour. If the process is cyclic and portions of the cycle give high particulate mass rates (PMR) which are not upset conditions, these portions of the cycle must be included in the sample. The sample size should be at least 850 liters at standard conditions. If conditions permit, some adjustments in nozzle size and sample flowrate can often be made to satisfy time requirements. It is usually best to sample each point for no less than three minutes as this allows time for adjusting flows and recording data.

### B.3.5.4 Sampling

When the console operator is ready and the probe and sample box have heated sufficiently, the initial dry gas meter reading is recorded and the sampling probe is pushed carefully into the duct to the point nearest the back wall. This allows the probe to cool in hot stacks as it comes out, shortening the time required for cooling after the sample is taken. This also allows the use of the stack heat to help heat the probe. The nozzle must not hit the back wall or the inside of the port where deposited material might contaminate the sample. If this procedure is followed, the last point sampled will be the point nearest the port. As soon as the probe is positioned the operator should record the time and start the run by turning on the pump, opening the coarse valve and adjusting the bypass valve until the desired flowrate calculated from the nomograph or K factor is obtained. The data should then be recorded. The probe crew should be notified 15 to 30 seconds before the probe is to be moved and the signal to move to the next point is given approximately 5 seconds before that time. When the probe is repositioned, the operator should read the new ΔP, use the nomograph or calculator, adjust the flow rate, and record the data required on sampling field data sheets. The opening at the port should be plugged to prevent dilution or abnormal distortion of the flow patterns in the stack. After the last point has been sampled, the operator turns off the pump and records the meter reading. If more than one port is to be sampled, the sampling box is transferred to the next port and the above procedure repeated. The probe is then removed carefully from the port so the open end of the nozzle does not hit the port. The probe should also be kept horizontal and the nozzle plugged as soon as possible after it is removed from the stack to prevent loss of sample. In some cases condensate will collect in the probe and if the probe is tipped, some of the sample might be lost. The particulate trapped in the probe can represent a significant portion of the total sample. Normally three sample runs are taken. Each run normally consists of two traverses. Each run is considered as a separate sample, and the calculations are performed for each sample. The results of the runs are then averaged.

Before making a second run the percent isokinetic of the first run should be checked. The nomograph or calculated

K factor should be changed and an additional sample taken if the percent isokinetic varies by more than ±10% from 100%. (See Data Reduction Section.)

### B.3.5.5 Sample Handling

At the end of each sample run the electrical power is disconnected and the hot side of the sample box is opened to allow the glassware to start cooling. The pitot tube lines to the probe are disconnected and the probe is removed from the box and the ends plugged as soon as possible. The impingers may now be removed and weighed. After the impingers are weighed, the liquid in the impingers is placed in clean sample bottles to be taken to the lab. Some particulate or condensable compounds will occasionally get past the filter and be collected in the liquid. The sample catch from the filter and the probe washings should be collected and stored separately from the impinger catch. The probe and nozzle are washed carefully and the washings collected in a clean sample bottle to be taken to the lab, evaporated and then weighed. The cyclone and flask and the connecting glassware are washed and the washings added to the probe washings. The weight of any water caught in the cyclone must be determined and added to the impinger weight gain. The filter is removed from the holder, folded with the particulate side in and placed in an envelope to be taken to the lab and weighed. The filter holder is washed and the washings added to the probe wash. Usually distilled water is used to wash the glassware, but in some instances, acetone may be used. Precautions must be taken to eliminate the possibility of tampering with, accidental destruction of, and/or physical and chemical action on the samples.

To reduce the possibility of invalidating the results, all components of the sample should be carefully removed from the sampling train and placed in sealed, nonreactive, numbered containers. The samples are then delivered to the laboratory for analysis. It is recommended that this be done on the same day that the samples are taken. If this is impractical, all samples should be placed in a carrying case (preferably locked) in which they are protected from breakage, contamination, and loss.

Each container should have a unique identification to insure positive identification and to preclude the possibility of interchange. The identification of the container should be recorded on the analysis data sheet so it will be associated with the sample throughout the test and analysis.

The samples should be handled only by persons associated in some way with the task. A good general rule to follow is "the fewer hands the better", even though a properly sealed sample may pass through a number of hands without affecting its integrity.

## B.4 MASS CONCENTRATION DATA REDUCTION

After performing particulate mass measurements on the inlet and outlet of an electrostatic precipitator, the data must be reduced to obtain particulate emissions concentrations and other pertinent parameters.

To illustrate these calculations, an example of data collected on a single test will be presented along with the appropriate calculations to obtain the necessary results.

From the particulate source test data given in Table B2 we find:

$$\text{Orifice } \Delta P_{avg} = 3.69 \text{ mm Hg}$$

# TABLE 41

## EXAMPLE - PARTICULATE SOURCE TEST DATA - ESP INLET

**General**

Firm - CM & S Public Service
Source - Coal Boiler #1
Sampling Location - ESP Inlet
Personnel - K.B.C., J.D.M., & W.B.S.
Date - 3/28/77
Test No.-5
Witnessed by - D.B.B.

**Equipment**

Gas Meter No.-3
Sample Box No.-4
Probe No.-7 Length-305 cm Cp-.87
Nozzle Dia. - 0.89 cm
Filter No. 222 Silica Gel No. 87
Probe Wash Sample No. - 4C13

**Conditions**

Orifice ΔH - 1.74 mm Hg
Assumed %H2O - 8% $T_m$ - 18°C
Assumed $\Delta P_{AVG}$ -1.5 mm Hg
Assumed $T_s$ -26.7°C - 4.29 m
Bar Pressure - 749 mm Hg
C-Factor - .862
Probe Heater Set - 135°C
Filter Oven Set - 122°C

| Traverse No. | Time (sec) | Dry Gas Meter ($x10^3 m^3$) | Pitot ΔP mm Hg | Orifice ΔH mm Hg | Meter Inlet °C | Temp. Outlet °C | Stack Temp. °C | Pump Vacuum mm Hg |
|---|---|---|---|---|---|---|---|---|
| 1 | 300 | 313.5 | .99 | 4.51 | 23 | 20 | 146 | 152.4 |
| 2 | 300 | 2446.8 | 1.09 | 4.89 | 24 | 20 | 146 | 152.4 |
| 3 | 300 | 4215.7 | 1.38 | 7.86 | 24 | 21 | 152 | 152.4 |
| 4 | 300 | 6577.4 | 0.85 | 4.35 | 24 | 22 | 150 | 177.8 |
| 5 | 300 | 9236.5 | 1.53 | 3.31 | 25 | 22 | 145 | 177.8 |
| 6 | 300 | 10866.3 | 1.42 | 3.16 | 25 | 22 | 147 | 152.4 |
| 7 | 300 | 13321.5 | 1.77 | 2.97 | 25 | 22 | 151 | 152.4 |
| 8 | 300 | 15613.4 | 1.76 | 2.80 | 25 | 22 | 151 | 152.4 |
| 9 | 300 | 19636.6 | 1.55 | 3.29 | 26 | 22 | 152 | 127.0 |
| 10 | 300 | 21137.8 | 1.36 | 3.33 | 27 | 23 | 150 | 152.4 |
| 11 | 300 | 23181.3 | 1.32 | 4.38 | 28 | 23 | 147 | 177.8 |
| 12 | 290 | 25060.1 | 1.38 | 3.74 | 28 | 24 | 145 | 177.8 |
| Totals | 3590 | 2600 | | | | | | |
| Averages | | | 1.28 | 3.89 | 25.3 | 21.9 | 148.4 | |

Stack Pressure -7.3 mm Hg

Test End - 12:03 noon
Test Start - 11:00 a.m.

Impinger Outlet Max Temperature 18°C

| $CO_2$ | $O_2$ |
|---|---|
| 11.2% | 6.2% |
| 11.5% | 5.5% |
| 11.3% | 5.6% |

Condensate Collected
#1 100 ml
#2 50 ml
#3 10 ml

Silica Gel Mass Change - +40 grams

$\Delta H_{AVG}$ = 1.64 mmHg

Filter and Probe Wash Catch - 150 mg

$$T_{m_{avg}} = 23.6°C = 296.9°K$$

### B.4.1 Volume of Gas Sampled

$$V_{mc} = V_{m_{std}} = V_m \frac{T_{std}(P_{bar} + \Delta H_{ave})}{T_m P_{std}}$$

$$= 2.8 \frac{294}{297} \frac{(749 + 3.69)}{760}$$

$$= 2.75 \text{ DNM}^3$$

where $V_{mc}$ is the volume of gas sampled through the dry gas meter at standard conditions (21°C, 760 mm Hg), DNM³.

$V_m$ is the volume of gas samples through the dry gas meter at meter conditions, m³.

$T_{std}$ is 21°C, 294°K.

$T_m$ is the average dry gas meter temperature, 23°C, 297°K.

$P_{bar}$ is the uncorrected barometric pressure at the outlet of the orifice meter, 749 mm Hg.

$\Delta H_{ave}$ is the average pressure drop across the orifice meter, 3.69 mm Hg.

$P_{std}$ is the absolute pressure at standard conditions, 760 mm Hg.

### B.4.2 Volume of $H_2O$ Vapor in Stack Gas

Water condensed in impingers = 160 ml, water absorbed on silica gel = 46 grams.

$$V_{wc} = V_{l_c} \frac{\rho_{H_2O}}{M_{H_2O}} \frac{R\, T_{std}}{P_{std}}$$

$$= 206 \frac{1.00}{18.0} \frac{(6.2383 \times 10^{4})(294)}{760}$$

$$= 0.28\ m^3$$

where $V_{wc}$ is the total volume of water in the sampled gas at standard conditions (21°C, 760 mm Hg), $m^3$.

$V_{l_c}$ is the total volume of liquid $H_2O$ collected in impingers and on silica gel, 206, ml.

$\rho_{H_2O}$ is the density of water at standard conditions, 1.00 g/ml.

R is the ideal gas constant, $6.2383 \times 10^{4}$ mm Hg $cm^3$/gm mole-°K.

$T_{std}$ is the standard temperature, 21°C, 294°K.

$P_{std}$ is the standard pressure, 760 mm Hg.

$M_{H_2O}$ is the molecular weight of water, gm/gm-mole.

B.4.3 Moisture Content of Stack Gas:

$$B_{wo} = \frac{V_{wc}}{V_{m_{std}} + V_{wc}}$$

$$= \frac{0.28}{2.75 + .28} = 0.0924 \text{ or } 9.24\%$$

where $B_{wo}$ is the mole fraction or proportion by volume of $H_2O$ vapor in the stack gas.

$V_{wc}$ is .28 m³ $H_2O$ vapor at standard conditions.

$V_{m_{std}}$ is 2.75 m³ of dry sampled gas at standard conditions.

### B.4.4 Molecular Weight of Stack Gas

From Orsat analysis per EPA Method 3:

$$\begin{gathered} CO_2 \text{ ave} = 11.5\% \\ O_2 \text{ ave} = 5.8\% \text{ by volume (dry basis)} \\ CO \text{ ave} = 0\% \end{gathered}$$

From moisture analysis per EPA Method 5:

$$H_2O \text{ avg} = 9.24\%$$

From EPA Method 3:

$$\% N_2 \text{ (dry basis)} = 100\% - (\% CO_2 + O_2 + \% CO)$$
$$= 100\% - (17.3\%) = 82.7\%$$
$$M_w = (1-B_{wo})(B_{CO_2}M_{CO_2} + B_{O_2}M_{O_2} + B_{CO}M_{CO} + B_{N_2}M_{N_2}) +$$
$$B_{wo}M_{H_2O}$$
$$= (1-.0924)\left[(0.115)(44.0) + (0.058)(32.0) + (0.0)(28.0) +\right.$$
$$\left.(0.827)(28.0)\right] + (0.0924)(18.0)$$
$$= 28.96 \text{ gm/gm-mole}$$

where $M_w$ is the molecular weight of the stack gas, gm/gm-mole, wet basis.

$B_i$ is the mole fraction of the component gas.
$M_i$ is the molecular weight of the component gas.
$B_{wo}$ is the mole fraction of the water vapor in the stack gas.

### B.4.5 Excess Combustion Air (per EPA Method 3)

$$\%EA = \frac{(\%O_2) - 0.5(\%CO)}{0.264(\%N_2) - (\%O_2 + 0.5(\%CO))} \times 100$$

$$= \frac{(5.8) - 0.5(0)}{0.264(83.7) - (5.8) - 0.5(0)}$$

$$= 36\%$$

where $\%EA$ is the percent excess combustion air.
$\%O_2$ is the percent $O_2$ by volume on a dry basis.
$\%N_2$ is the percent $N_2$ by volume on a dry basis.
$\%CO$ is the percent CO by volume on a dry basis.
0.264 is the ratio of $O_2$ to $N_2$ in air by volume.

### B.4.6 Particulate Emissions Concentration

$$C'_s = \frac{M_n}{V_{m_{std}}}$$

$$= \frac{150.0}{2.75} = 54.55 \text{ mg/DNM}^3$$

where $C'_s$ is the concentration of particulate matter in the stack gas, mg/DNM³.
$M_n$ is the total particulate mass collected on filter media, 150.0 mg.
$V_{m_{std}}$ is the volume of stack gas sampled (volume through dry gas meter), DNM³.

B.4.7 Stack Gas Volumetric Flow Rate (per EPA Method 2)

$$(V_s)_{avg} = K_p C_p (\sqrt{\Delta p})_{avg} \left[\frac{(T_s)_{avg}}{P_s M_w}\right]^{1/2}$$

where $(V_s)_{avg}$ is the average stack gas velocity, m/sec.

$K_p$ is $128.81 \frac{m}{sec}\left(\frac{gm}{gm\text{-}mole\text{-}°K}\right)^{1/2}$

$C_p$ is the pitot tube coefficient.

$(T_s)_{avg}$ is the average stack gas temperature, °K.

$(\sqrt{\Delta p})_{avg}$ is the average of the square roots of velocity heads of stack gas, in $H_2O$, determined according to EPA Methods 1 and 2.

$P_s$ is the absolute stack gas pressure, mm Hg.

$M_w$ is the molecular weight of the stack gas, gm/gm-mole (wet basis).

From Table B2:

$$(\sqrt{\Delta p})avg = 1.13$$

$$C_p = 0.860$$

$$(T_s)avg = 421.7\ °K$$

$$P_s = P_{bar} + (P_s) = 749 + (-.75)$$

$$= 748.25 \text{ mm Hg}$$

$$M_w = 28.96 \text{ gm/gm-mole}$$

$$(V_s)_{avg} = (128.83)(0.860)(1.13)\left[\frac{421.7}{(748.25)(28.96)}\right]^{1/2}$$

$$(V_s)_{avg} = 17.47 \text{ m/sec}$$

Then calculating the average stack gas volumetric flow rate:

$$\bar{Q}_s = 3600 \ \frac{sec}{hr} \ (1-B_{wo})(V_s)_{avg} \ A \ \frac{T_{std}P_s}{(T_s)_{avg} \ P_{std}}$$

where $\bar{Q}_s$ is the average volumetric flow rate at dry standard conditions, DNM$^3$/h

$B_{wo}$ is the mole fraction of water vapor in the stack gas, dimensionless, and

$A_s$ is the cross-sectional area of the stack at the sample point, 9.29 m$^2$ (from the Particulate Test Data Sheet).

Then,

$$\bar{Q}_s = (3.600 \times 10^3)(.9076)(17.47)(9.29) \frac{(294)(748.25)}{(421.7)(760)}$$

$$= 3.64 \times 10^5 \text{ DNM}^3/\text{h}$$

Therefore, the particulate inlet mass emission rate can be calculated by $\overline{PMR}_s = \bar{Q}_s \ c_s$

where $\overline{PMR}_s$ is the average particulate inlet mass emission rate, (mg/hr calculated by the concentration method).

$$\overline{PMR}_s = 3.64 \times 10^5 \ \frac{\text{DNM}^3}{\text{hr}} \cdot 54.55 \text{ mg/DNM}^3$$

$$= 1.986 \times 10^7 \text{ mg/hr of particulate.}$$

### 8.4.8 Average Isokinetic Ratio

$$I_{avg} = \frac{(V_n)_{avg}}{(V_s)_{avg}} \times 100$$

where $I_{avg}$ is the average percent isokinetic.

$(V_n)_{avg}$ is the average gas velocity into the nozzle entrance.

$(V_s)_{avg}$ is the average gas velocity of the stack gases.

Writing the equations for the nozzle velocity independent of the velocity pressure measured in the stack.

$$(V_n)_{avg} = \frac{V_{mc} \dfrac{T_s P_m}{T_m P_s M_d}}{A_n \theta}$$

where $(V_n)_{avg}$ is the average nozzle velocity.

$V_m$ is the volume of gas through the dry gas meter, DNM³ (dry).

$T_s$ is the average absolute stack gas temperature °K.

$P_s$ is the average absolute stack pressure, mm Hg.

$P_m$ is the average absolute dry gas meter pressure, mm Hg

$$(P_m = \Delta m + P_{bar})$$

$T_m$ is the average absolute meter temperature, °K

$M_d$ is the average mole fraction of the dry stack gas, $(1-B_{wo})$.

$A_n$ is the area of the nozzle, m².

$\theta$ is the total sampling time, sec.

Then,

$$(V_n)_{avg} = \frac{(2.8)\ \frac{421.7}{294}\ \frac{752.69}{748.21}\ \frac{1}{.9076}}{(6.22 \times 10^{-5} m^2)\ (3600\ sec)}$$

$$= 19.88\ m/sec$$

$$I_{avg} = \frac{(V_n)avg}{(V_s)avg} = \frac{19.88}{17.47} = 1.14 \text{ or } 114\%$$

To conform with the federal performance standards, the average isokinetic rate $I_{avg}$, must be $\geq 0.90$ and $\leq 1.10$. In this example, since $I_{avg}$ does not fall within these limits, this test would be discarded.

### 8.4.9 Mass Collection Efficiency Calculation

This example has shown calculations for a test at a precipitator inlet. Similar calculations are made on data taken at ESP outlets. After determining the inlet and outlet mass loading concentrations, the precipitator efficiency can be calculated from

$$\%EFF = \frac{PMR_{Inlet} - PMR_{Outlet}}{PMR_{Inlet}} \times 100\%$$

$PMR_{Inlet}$ = Particulate inlet mass emission rate.

$PMR_{Outlet}$ = Particulate outlet mass emission rate.

# APPENDIX C

# CASCADE IMPACTOR SAMPLING TECHNIQUES

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## APPENDIX C
## CASCADE IMPACTOR SAMPLING TECHNIQUES

This appendix contains detailed guidelines for the operation of cascade impactors to measure the particle size distributions at the inlet and outlet of electrostatic precipitators. These instructions are taken from "PROCEDURES FOR CASCADE IMPACTOR CALIBRATION AND OPERATION IN PROCESS STREAMS", EPA-600/2-77-004, by D. B. Harris. Minor modifications were made to make them specifically applicable to precipitator evaluation, and a more detailed description of data reduction techniques and data presentation formats has been added.

### C.1 THE PRESURVEY

The key to performing a successful fractional efficiency evaluation is thorough planning based on a complete pretest site survey. The survey should provide adequate information at as low a cost as possible. Some sites will require more information and some less. As far as is possible, the information noted during the presurvey should be measured rather than obtained from plant records or personnel.

As the presurvey is generally conducted by one or two men "traveling lightly," the apparatus used during the presurvey should be as light and compact as possible. A presurvey sample train is shown in Figure C1. This system can be built into a single, suitcase-size package and serves well as a presurvey sample train. The impactor which is to be used during the main test program should normally be used during the presurvey. This is because the suitability of substrates and adhesives must be checked out. These problems are discussed more fully in later sections.

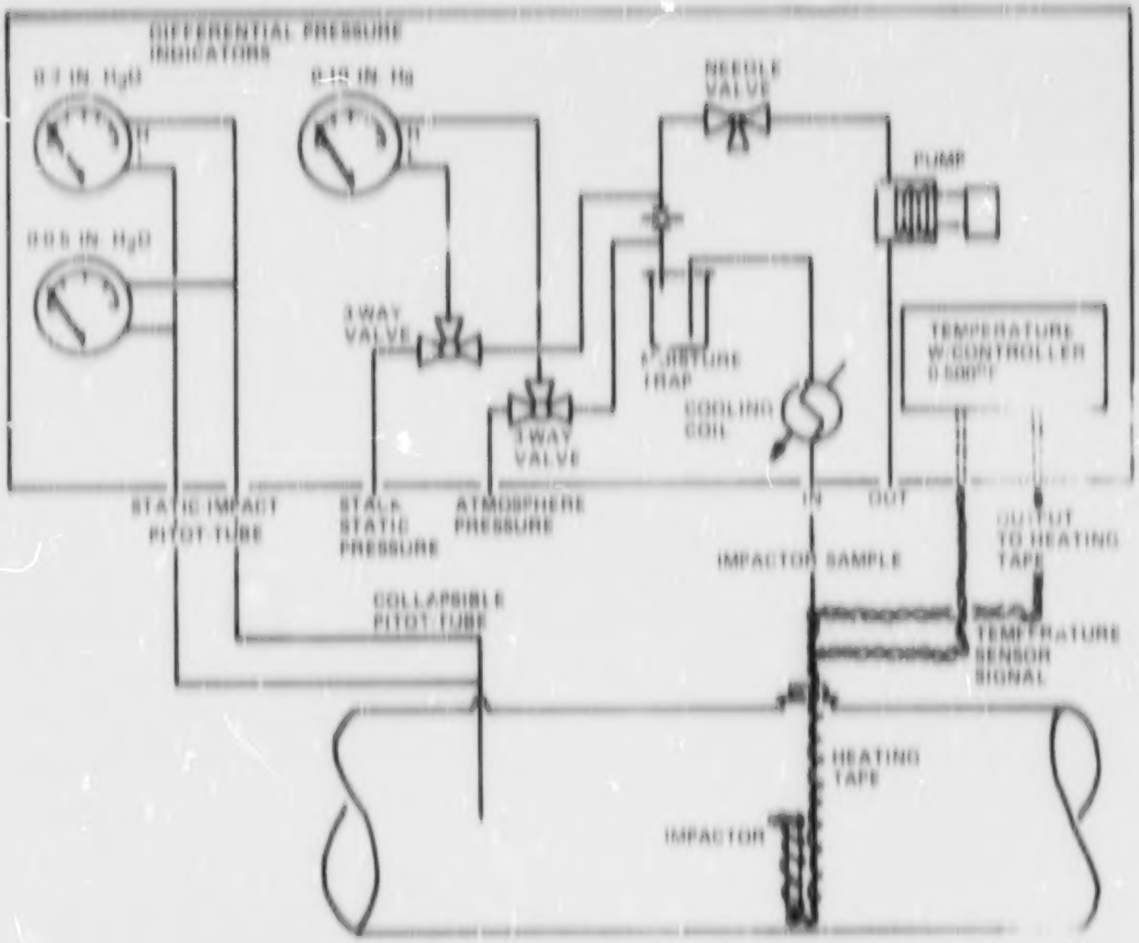
DIFFERENTIAL PRESSURE INDICATORS
0-7 IN. H2O
0-15 IN. Hg
0-0.5 IN. H2O
NEEDLE VALVE
PUMP
3 WAY VALVE
MOISTURE TRAP
3 WAY VALVE
COOLING COIL
TEMPERATURE W/CONTROLLER 0-500°F
STATIC IMPACT
PITOT TUBE
STACK STATIC PRESSURE
ATMOSPHERE PRESSURE
IN
OUT
OUTPUT TO HEATING TAPE
IMPACTOR SAMPLE
COLLAPSIBLE PITOT TUBE
TEMPERATURE SENSOR SIGNAL
HEATING TAPE
IMPACTOR

*Figure C1. Presurvey sampling with a cascade impactor.*

In general, the presurvey work should be done using the techniques described in this guideline. Less precision is required, but the accuracy must be high enough to provide useful information in designing the test program. The decisions which must be made are summarized in Table C1.

## C.2 EQUIPMENT SELECTION

### C.2.1 Impactor Selection

The selection of the proper impactor for a particular test situation is primarily dependent upon the mass loading of the gas stream and its effect on sampling time. There are three major criteria to be met to match an impactor to a particulate stream.

1. The sampling period must be long enough to provide a reasonable averaging of any short term transient in the stack.

2. The loading on a given impactor stage must be low enough to prevent re-entrainment.

3. The sampling rate through the impactor must be low enough to prevent scouring of impacted particles by high gas velocities.

For these reasons, an impactor with a comparatively low sample rate must be used in a gas stream with a high mass loading. The low sample rate allows a longer sampling time, although in some situations it will still be undesirably short. Conversely, in a low mass loading situation such as a control device outlet, a high sample rate device must be used if a significant amount of sample is to be gathered in a reasonable amount of time.

## TABLE C1. IMPACTOR DECISION MATRIX

| Item | Information Required | Criteria |
|---|---|---|
| Impactor | Loading and size estimate | a. If particle concentration below 5.0 µm is less than 0.46 $gm/am^3$ (0.2 grain/acf), use high flow rate impactor. |
| | | b. If particle concentration below 5.0 µm is greater than 0.46 $gm/am^3$ (0.2 grain/acf), use low flow rate impactor. |
| Sampling rate | Loading and gas velocity | a. Fixed, near isokinetic |
| | | b. Limit so last jet velocity does not exceed:<br>60 m/sec greased<br>35 m/sec without grease |
| Nozzle | Gas velocity | a. Near isokinetic, ±10% |
| | | b. Sharp edged; min 1.4 mm ID |
| Pre-cutter | Size and loading | If pre-cutter loading is comparable to first stage loading—use pre-cutter. |
| Sampling time | Loading and flow rate | a. Per Figure C4 |
| | | b. No stage loading greater than 10 mg |
| Collection substrates | Temperature and gas composition | a. Use metallic foil or fiber substrates whenever possible |
| | | b. Use adhesive coatings whenever possible |
| Number of sample points | Velocity distribution and duct configuration | a. At least two points per station |
| | | b. At least two samples per point |

A cascade impactor can normally yield useful information over a range of sample rates differing by a factor of 2 or 3. As high efficiency control devices cause the outlet mass loading to differ from the inlet by a factor of $10^3$, the same impactor can seldom be used on both inlet and outlet. Both high and low flow rate impactors are usually required to determine the fractional efficiency of electrostatic precipitators. Some commercial impactors are constructed such that their stage and nozzle configurations can be altered, and they can serve as either high or low sample rate impactors. Others are fixed with respect to sample rate.

### C.2.2 Sample Trains

Figure C2 is a flow diagram of a typical impactor sample train. As shown, it is desirable to have the impactor inside the stack with a straight nozzle. The various parts of the sample train are discussed below.

A sampling probe leading to an impactor outside of the duct should be used only if absolutely necessary. The probe should be as short as possible and contain the fewest possible bends. It is recommended that a pre-cutter cyclone be mounted at the probe inlet to remove particles larger than approximately ten micrometers and thus reduce line losses.

Heating system -- The criteria for heating are given in Section C.4.3. If heating is required, the entire impactor must be either wrapped in a heating tape or put in a custom-fitted heating mantle. The temperature control should be based on the temperature at the outlet end of the impactor. Often the temperature is measured between the last stage and back-up filter. The impactor temperature can be controlled either manually or automatically. An automatic con-

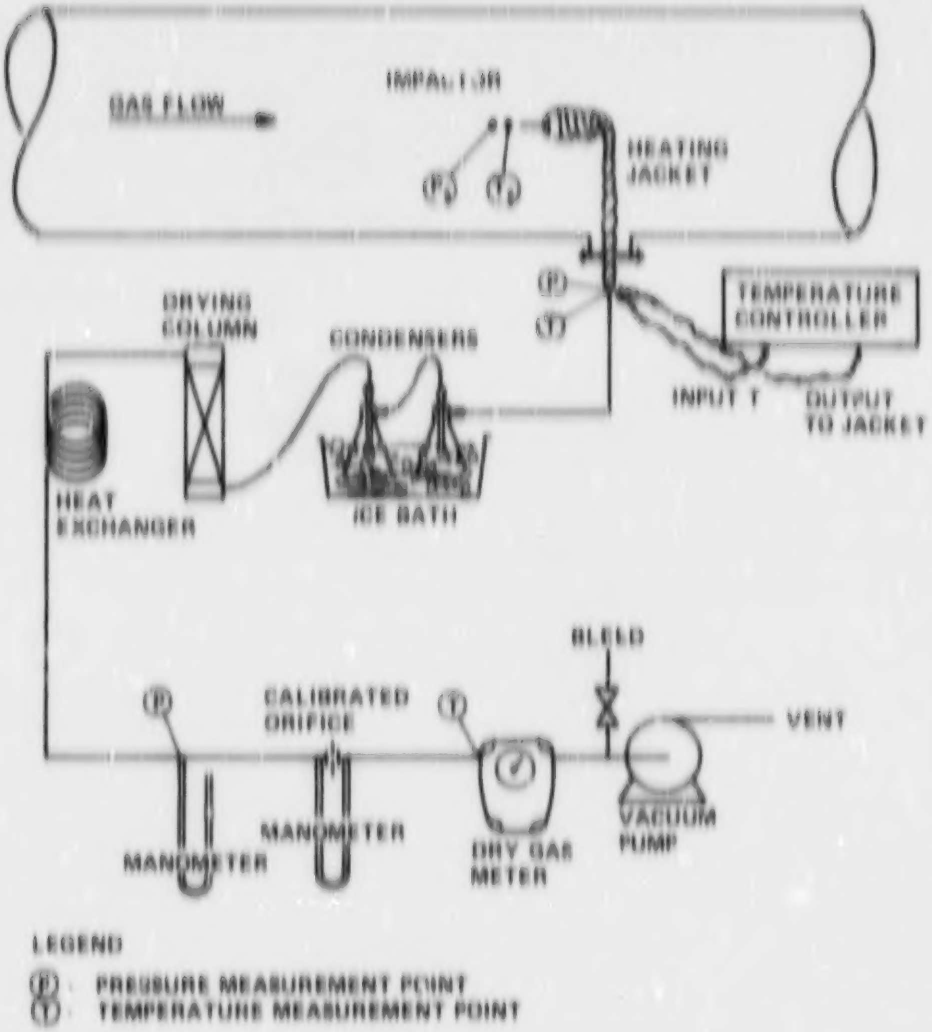
GAS FLOW
IMPACTOR
$P_i$
$T_i$
HEATING JACKET
P
T
TEMPERATURE CONTROLLER
INPUT T
OUTPUT TO JACKET
DRYING COLUMN
CONDENSERS
HEAT EXCHANGER
ICE BATH
BLEED
VENT
P
CALIBRATED ORIFICE
T
MANOMETER
MANOMETER
DRY GAS METER
VACUUM PUMP
LEGEND
P · PRESSURE MEASUREMENT POINT
T · TEMPERATURE MEASUREMENT POINT

*Figure C2. Typical sample train with a heated impactor.*

troller has usually been found to be worth the money by releasing the operator for other tasks.

*Flue gas conditioning* -- It is usually necessary to cool and dry the flue gas before it reaches the flow measuring section. Condensation in the orifice would distort the measurement; also, it is useful to protect the equipment from the condensate, which, in $SO_2$-containing gases, is likely to be sulfuric acid. The type of condensers shown are usually satisfactory. Packed bed drying columns are commercially available. The heat exchange coil is used to bring the gas temperature to essentially ambient so that there will not be a significant temperature gradient across the flow measuring devices.

*Flow measurements* -- At least two flow measuring devices are used in series. Normally, a calibrated orifice is used in conjunction with a dry gas meter, as shown. At very low sample rates the dry gas meter may be inaccurate. The commonly used diaphragm-type positive displacement gas meter becomes increasingly inaccurate at flow rates less than five percent of rated capacity. For a typical stack sampling gas meter this would be approximately 23.3 $cm^3$/sec (0.05 cfm). Another calibrated orifice or a rotameter should then be used as the second flow meter.

*Vacuum pumps* -- The vacuum pump should usually be placed at the end of the sample train. This is because vacuum pumps tend to leak and all of the flow measurements must be made upstream of any leak. The flow rate can be controlled by using an inlet side air bleed or with a recirculating bypass.

Pressure measurements -- Most of the pressure measurements are made with manometers, but calibrated differential pressure meters are equally acceptable. The in-stack pressure needed is the static pressure, which is not exactly the downstream pressure of an S-type pitot tube. A true static pressure measurement should be made. It is not necessary that this be part of the impactor train, but it can be.

The pressure at the downstream end of the impactor, between the last stage and the final filter, must be known. It can be measured, but this is often inconvenient. If a flow rate pressure drop calibration is available for the impactor (without final filter), it is normally acceptable to calculate the pressure drop. Correction must be made for pressure and temperature differences between the calibration conditions and the actual conditions.

The pressure at the inlet to the metering devices must be known. In the system shown in Figure C2, the pressure is metered ahead of the calibrated orifice and the orifice pressure drop is used to calculate the pressure going into the dry gas meter. The dry gas meter pressure should be measured if there is a reason to think the procedure above was not adequate.

Temperature measurements -- It is necessary to know the temperature at all points where the pressure is measured. Any convenient device of known accuracy can be used to make the measurements. The measurement in-stack can easily be made at the probe end with a thermocouple. The temperature at the downstream end of the impactor is made directly behind the final filter and is used to control the heating tape if one is used. If the heat exchanger in the train brings the gas temperature to about ambient, only one temperature reading will be necessary at the flow meters. This is usually most conveniently done at the dry gas meter, as taps are available on the meter.

### C.2.3 Balance Requirements

For the accurate weighing of collected material a balance with a sensitivity of at least 0.05 mg is required. This is especially true for the lower stages of the low sample rate impactors where collection of 0.3 mg or less is not uncommon. The balance must also be insensitive to vibration if it is to be used in the field. It is also desirable to have a balance with a large weighing chamber. These capabilities are available in several electrobalances marketed in the United States.

More information on selected sampling train components can be found in Appendix B, Section B.1.

## C.3 IMPACTOR SUBSTRATES

### C.3.1 Collection Substrates

For reasons which have been discussed, very accurate determinations of impactor stage catch weights are necessary. Impactor stages are generally too heavy for the tare capacity of field-usable precision balances. Thus, a substrate which can be weighed on the balance is used. Generally, these substrates are made of metal foil or glass fiber.

Glass fiber substrates -- Glass fiber substrates are used on some commercial impactors. In addition to providing a lightweight impaction surface, glass fiber mats greatly reduce re-entrainment due to particle bounce. They are superior to greased metal substrates in very high temperature applications where the greases tend to evaporate. Care must be taken when using glass fiber substrates in streams containing sulfur dioxide, however. Recent experimentation[1] has shown that glass fiber materials often exhibit anomalous weight gains due to sulfate on the substrates. Apparently, sulfur dioxide in a gas stream can react with basic sites on most glass fiber materials and form sulfates.

There are two approaches to alleviate this problem. Substrates which will gain weight from sulfate uptake can be preconditioned in the flue gas before weighing. Two to six hours of exposure to the flue gas will suffice where mass loadings are high and sample times are short. In the situations where sample times are long and the collected amount of particulate matter is small, it may be necessary to condition the substrates for as long as twenty-four hours to eliminate significant sulfate uptake and weight gains. Repeated weighings to check weight gains are necessary to confirm that the substrates can be used. Another approach is to use a fibrous substrate which shows little weight gain in a sulfur dioxide stream, if one can be found. It should be noted that the particle retention characteristics of different fiber materials vary, and the impactor calibration could change significantly if the substrate is changed.

Greased substrates -- "Grease" must often be used on metal foil substrates to improve their particle retention characteristics. This is particularly important with hard, bouncy particles. Impactor stage velocities of 60-65 m/sec have been used on greased substrates with good results, while particle bounce can become a problem at about half of that rate on ungreased substrates.

Finding a suitable grease can be difficult. The grease should not flow at operating temperature, and it must be essentially nonvolatile. Gas chromatographic materials such as polyethylene glycol 600 have exhibited more consistent characteristics than materials such as stopcock grease. Another class of materials which may be suitable are high vacuum greases; Apiezon L and H in particular have performed well at temperatures up to 120°C. The greased substrates must be tested as blanks in filtered process gas before they are used in the test program.

The greases are normally applied as suspensions or solutions to 10-20 percent grease in toluene or benzene. The mixture is placed on the substrate with a brush or eyedropper, baked at 204°C for 1 to 2 hours, and then desiccated for 12 to 24 hours prior to weighing. It is important to avoid an excess of grease. The desiccated, greased substrate should be tacky, but not slippery, with a film thickness about equal to the diameter of the particles which are to be captured.

Horizontal operation of the impactors with greased substrates is not recommended due to possible flow of the grease. Care must also be taken to ensure that grease is not blown off the substrates (which tends to occur at jet velocities greater than 60 m/sec). To some degree, grease blow-off can be avoided by using a light coating of grease on the last stages. This is normally satisfactory from an adhesive standpoint, as the last stages usually have the lightest loading along with the highest jet velocity. Inspection of the stage catches is the best way to check on this problem.

### C.3.2 Back up Filters

Back up filters are used on all impactors to collect the material that passes the last impaction stage. Binderless glass fiber filter material is normally used for this purpose in all the impactors, although the exact configuration varies.

Glass fiber back up filters have the same problems as do glass fiber substrates. Their use in process gases containing sulfur dioxide is suspect, and blanks must be run to check out the problems. Pure Teflon filters may alleviate this problem if they can be used.

## C.4 PREPARATION AND SAMPLING

### C.4.1 Substrate Preparation

It is assumed that the substrates have been properly prepared and that the necessary quality assurance steps have been taken. The substrates should be carefully weighed and kept in a desiccator until they are to be placed in the impactor.

### C.4.2 Impactor Orientation

Whenever possible, the impactor should be oriented vertically to minimize gravitational effects such as flow of grease or fall-off of collected particles. Sampling situations requiring horizontal placement will occur, and extra care must be taken on such occasions not to bump the impactor against the port during entry or removal.

### C.4.3 Heating the Impactor

All condensable vapors must be in [illegible]ous state until they exit from the impactor, unless a [illegible]te is the prime aerosol being measured. In gas streams [illegible] 177°C, auxiliary heating should not be required. Below 177°C the exit temperature of the impactor should be maintained at least 10°C above the process temperature if condensable vapors are present. A thermocouple feedback temperature controller has proven useful.

When condensable vapors are present, it is sometimes necessary to heat the impactor probe to prevent any condensate formed in the probe from entering the impactor and contaminating the substrates. Water vapor is the primary problem. The probe temperature should be maintained above the vapor's dewpoint.

Whether the impactor is being heated in the duct or externally with heater tape, an allowance of 45 minutes warmup time is recommended as a minimum to ensure that the impactor has been heated to duct or operating temperature. Thermocouple monitoring of the impactor temperature and gas temperature is recommended.

### C.4.4 Probes

Sampling probes leading to an impactor outside of the duct should be used only if there is no other way. They should be as short as possible and contain the fewest possible bends. It is recommended that a precutter be mounted at the duct end of the probe to remove the large (>10 um) particles and thus reduce line losses.

### C.4.5 Nozzle and Sampling Rate Selection

It is preferable to use as large a nozzle diameter as possible to minimize sampling errors resulting from nozzle inlet geometry. When very small nozzles have been used with the Brink impactor, there have been some cases in which large amounts of material were retained in the nozzle or the nozzle was completely blocked. It is recommended that the inlet nozzle not be smaller than 1.4 mm, and some types of particulate material may require a larger minimum nozzle size. In some instances bent nozzles are necessary due to port location and gas direction, but these should be avoided. Problems occur in cleaning bent nozzles, and it is difficult to determine the size interval in which the deposited material originated. If they cannot be avoided, bends should be as smooth as possible and of minimum angle in order to minimize the losses in the fine particle region.

For hard, "bouncy", particulate, the sampling rate must be such that the last stage velocity does not exceed 60 m/sec for greased collection surfaces or 35 m/sec for ungreased plates if no suitable substrate can be found to limit particle bounce. The flow rates above should not be considered the final word on nozzle velocity. Particle bounce has been observed at nozzle velocities as low as 10 m/sec. Some particulate materials are "sticky" and will adhere at well above the maximum velocity for hard particles. The exposed substrate should be visually examined for evidence of re-entrainment and the rates adjusted accordingly.

It is apparent that sample rate and nozzle size are closely coupled. The requirements for isokinetic or near-isokinetic nozzle flow sometimes impose a compromise on nozzle selection. The general order of priorities when choosing the sample rate is nozzle diameter (at least 1.4 mm), last stage jet velocity and flow rate required for isokinetic sampling. Selection of nozzle diameter and impactor flow rate combinations for achieving near-isokinetic sampling conditions can be made from Figure C3.

If a choice must be made between undersized and oversized nozzles, undersized nozzles will usually result in lower sampling errors than will oversize.

### C.4.6 Use of Precollector Cyclones

In many instances the percentage (by weight) of material with sizes larger than the first impaction stage cut point is quite high. In such cases a precollector cyclone is necessary to prevent the upper impactor stages from overloading. A precutter should always be used for the first test. If the weight of material obtained by the precutter is greater than or equal to that on the first stage, the precollector should be used in all subsequent runs. Cyclones can be obtained from the impactor manufacturer or can be shop made. The use of two first stages in series has also been suggested and appears to be a valid approach; however, no data are available.

### C.4.7 Impactor Flow Rate Measurement

The flow rate through an impactor must be accurately measured in order to set the isokinetic sampling rate and to determine the correct impactor stage cut points. Unfortunately, it is usually very inconvenient, and sometimes

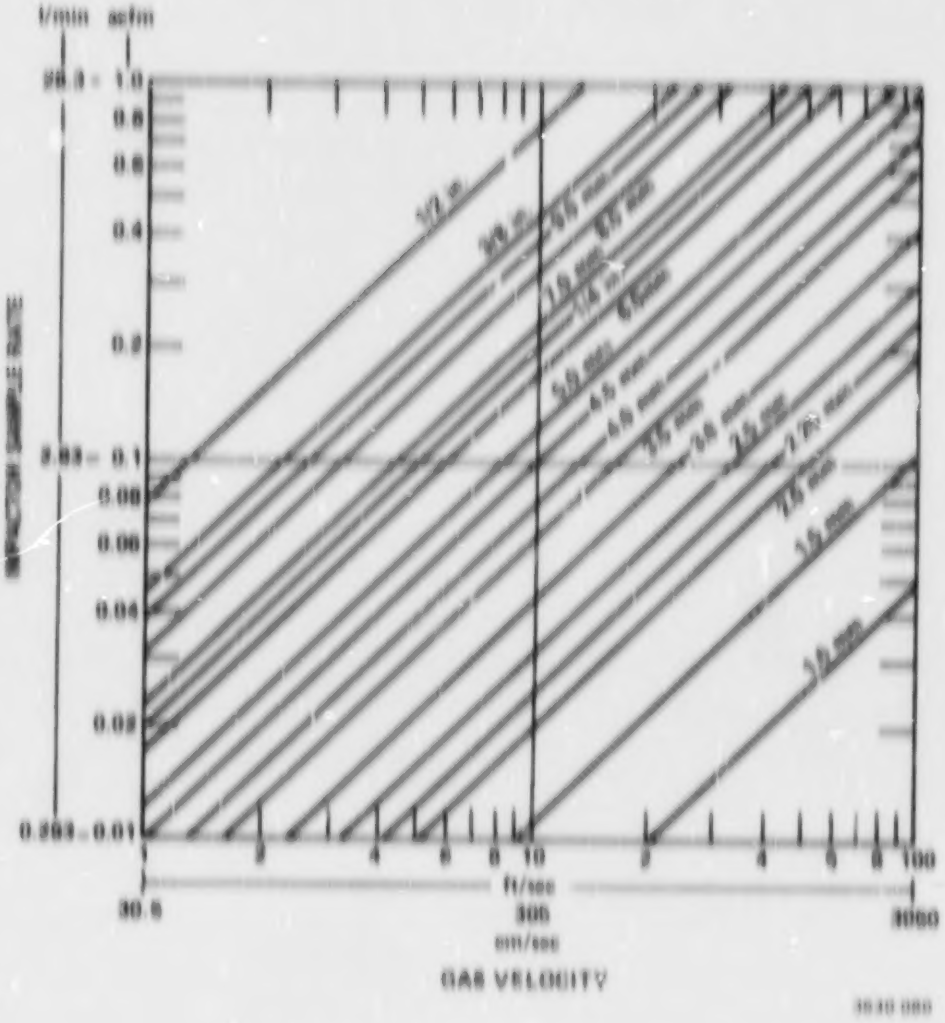

*Figure C3. Nomograph for selecting nozzles for isokinetic sampling.*

impossible, to measure the impactor flow rate at the conditions present in the impactor. The gas is normally drier, cooler, and at a lower pressure by the time the flow rate is measured; and the flow must be corrected to impactor conditions. The use of calibrated orifices and dry gas meters is discussed below.

Units -- The equations presented are valid only if the units of the various terms are consistent. For instance, the pressure drop terms could be in units of mm $H_2O$ or cm $H_2O$ or something else, but all pressure drop terms must have the same units. The same is true for the other properties. Note that pressure and temperature are both absolute measurements.

Orifice meters -- The gas flow rate through a particular orifice meter is related to the pressure drop across that orifice by an equation of the form:

$$Q^2 = C \frac{\Delta P}{\rho} \qquad \text{(C1)}$$

where $Q$ = volumetric flow rate at upstream conditions,
$\Delta P$ = pressure drop across orifice,
$\rho$ = density of gas at upstream conditions, and
$C$ = dimensional constant, $(\text{length})^5(\text{mass})(\text{time})^{-2}(\text{force})^{-1}$.

Solving for the constant, C, in equation (C1), one obtains:

$$C = \frac{Q^2\rho}{\Delta P} \qquad \text{(C2)}$$

As C is a constant at all conditions, its value can be obtained at a convenient set of conditions with a known flow rate and used later to calculate the flow rate. Equation (C2) can be rewritten to:

$$C = \frac{Q_c^2 \rho_c}{\Delta P_c} \qquad \text{(C2a)}$$

The subscript "c" indicates that these parameters were determined during a calibration. Density and flow rate are at upstream conditions.

Substituting equation (C2a) into equation (C1) yields an equation suitable for obtaining flow rates from a calibrated orifice:

$$Q_m^2 = \left[\frac{Q_c^2 \rho_c}{\Delta P_c}\right] \frac{\Delta P_m}{\rho_m} \quad , \qquad \text{(C3)}$$

The subscript "m" denotes the parameters of the gas as it is being "measured". All are at conditions immediately upstream of the orifice.

For use with impactors, the measured flow rate, $Q_m$, must be converted to a flow rate at stack conditions, $Q_s$. Assuming that the stack gas was dried as well as altered in temperature and pressure, the stack flow rate is related to the measured flow rate by:

$$Q_s(1 - B_{wo})\frac{P_s}{T_s} = Q_m \frac{P_m}{T_m} \qquad \text{(C4)}$$

where $B_{wo}$ = water removed from flue gas, expressed as a volumetric fraction,

$P$ = absolute pressure, and

$T$ = absolute temperature.

The subscript "s" refers to stack conditions.

At the usual conditions of relatively high temperature and low pressure which occur during stack sampling, the flue gas behaves very much like an ideal gas. The density of an ideal gas can be approximated as:

$$\rho = \frac{P(MW)}{(R)(T)} \qquad (C5)$$

where R = the universal gas constant, and
MW = the molecular weight of the gas.

Equations (C3), (C4), and (C5) can be combined and rearranged into a form which gives the pressure drop which must exist across the calibrated orifice, $\Delta P_m$, to obtain the required impactor flow rate, $Q_s$.

$$\Delta P_m = \Delta P_c \frac{Q_s^2}{Q_c^2} (1-B_{wo})^2 \left(\frac{P_s^2}{P_m P_c}\right)\left(\frac{T_m T_c}{T_s^2}\right)\left(\frac{MW_m}{MW_c}\right) \qquad (C6)$$

where $(MW)_m$ = molecular weight of the stack gas at the orifice, normally the dry molecular weight,
$(MW)_c$ = molecular weight of the calibration gas.

Dry gas meter -- The dry gas meter, like the orifice, can only directly measure the flow rate of the gas which passes through it. This measured flow rate can be converted to the flow rate through the impactor (which is at stack conditions) using equation (C4a):

$$Q_m = Q_s \left(\frac{T_m}{T_s}\right)\left(\frac{P_s}{P_m}\right) (1 - B_{wo}) \qquad (C4a)$$

## C.4.8 Sampling Time

The length of the sampling time is dictated by mass loading and size distribution. An estimate for initial tests can be obtained from Figure C4. Two conflicting criteria complicate the choice of the sampling time. It is desirable from the standpoint of minimizing weighing errors to collect several milligrams on each stage. However, most size distributions are such that the upper stages are overloaded and are re-entraining particles by the time the lower stages reach a few milligrams. A rule of thumb is that no stage should be loaded above 10 mg, but the determining factor is whether or not re-entrainment occurs. As is discussed later, a comparison of the relative distribution determined by a long run with that from a shorter (about half as long) run can be used to check on re-entrainment due to stage overloading.

## C.4.9 Readying the Impactor

As equipment is not always cleaned as well as it should be, the impactor should be inspected prior to use. The nozzles must be clean, gaskets in good shape, and the interior clean. Nozzles can be cleaned with fine wire if necessary.

After inspection, the impactor should be carefully loaded with the preweighed stage substrates and assembled. Teflon thread sealant tape or antiseize compound should be applied to the threads, especially when high temperatures (>215°C) are encountered. The thread sealant tape generally works better and causes fewer problems but probably cannot be successfully used at temperatures above 290°C.

If supplemental heating is required, a heating device and temperature monitor need to be added. A thermocouple

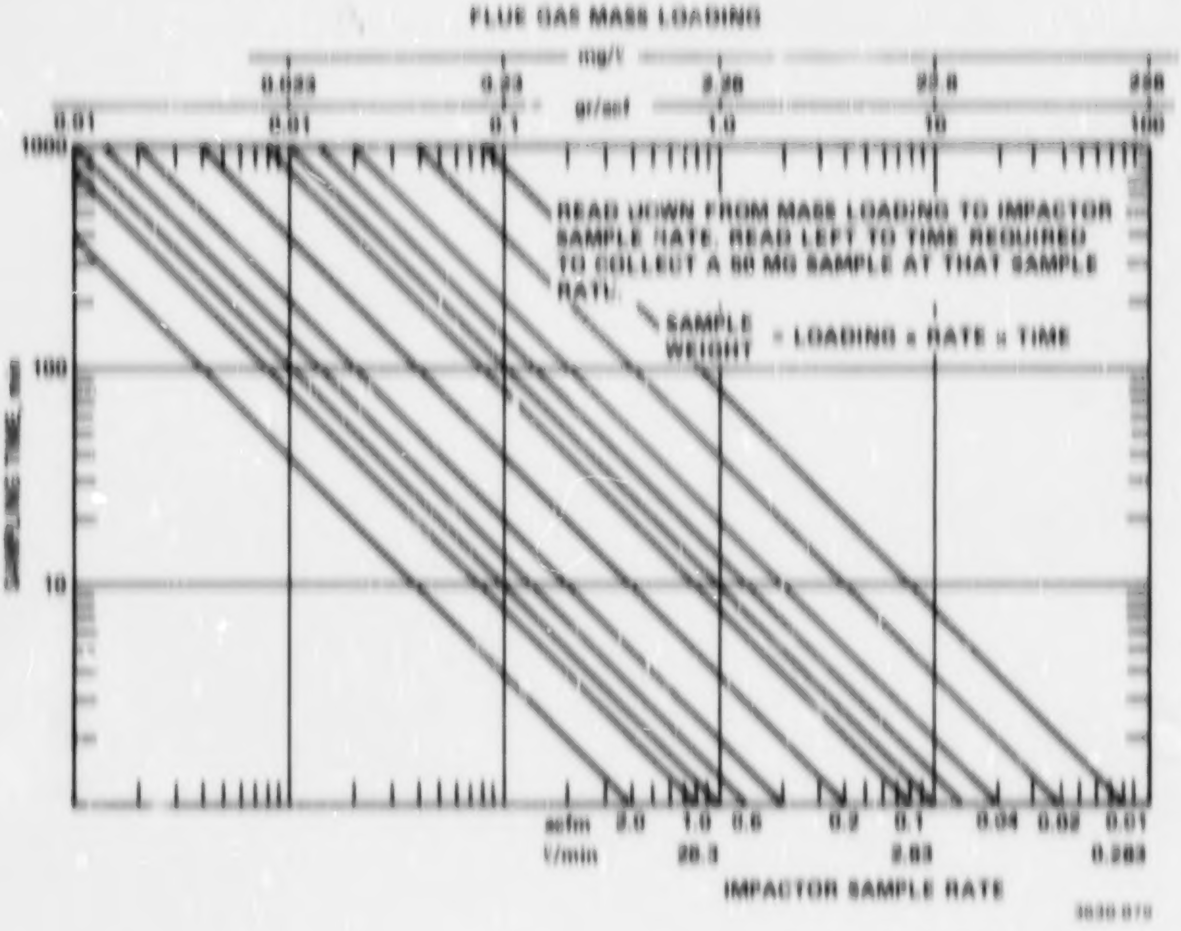

*Figure C4. Nomograph for sampling time selection (50 mg sample).*

mounted in the gas flow immediately after the impactor is best for controlling impactor cut points.

The supplemental heat can be supplied with either a heating mantle which has been made to fit the impactor or by using heating tapes. If the tapes are to be used, a heating tape of sufficient wattage is wrapped around the impactor. Glass fiber tape works well for holding the heating tape. Insulation such as asbestos tape is then wound around the impactor. Glass fiber tape again is used to hold the asbestos in place and also acts as additional insulation. The impactor can now be mounted on the appropriate probe, taken to the sampling position, and installed in the sampling system.

### C.4.10 Pre-Sample Checks

Impactors are prone to leak, and they must be checked at operating temperature for leaks. This can be done in several ways. The nozzle can be plugged and the impactor pressure-tested or vacuum-tested. Because impactors are basically a series of orifices, they should have a constant flow to pressure drop relationship. Checking the pressure drop on various flows of filtered air will point out deviations from normal operations--both leaks (external or internal) and plugged jets.

### C.4.11 Taking the Sample

The impactor should be preheated for at least 45 minutes before sampling. If supplemental heat is being used, the impactor should be brought up to temperature outside the duct and then allowed some time to equilibrate after insertion.

The nozzle should not point into the flow field during this phase. Without supplemental heat, the whole warm-up is conducted within the duct, again with the nozzle pointed away from the flow field.

A predetermined flow rate must be maintained to ensure stable cut points. Any attempt to modulate flow to provide isokinetic sampling will destroy the utility of the data by changing the cut points of the individual stages. Rapid establishment of the correct flow rate is especially important for the short sampling times typically found at the inlets to control devices. Capping the nozzle during preheat in the flue is also desirable.

### C.4.12 Number of Sample Points

As the velocity and particulate distributions in industrial ductwork are unlikely to be ideal, a large number of samples are often required for accurate particulate measurements. A velocity traverse should be run to check on the velocity distribution. At least two points within a duct should be sampled in each measurement plane, and at least two samples taken at each of these points. These are the minimum sampling efforts and are appropriate only for locations with well developed flow profiles in the absence of significant concentration stratification. If the flow profile at the inlet or outlet is uncertain due to duct configuration and/or the mass loading is not uniform, the number of samples may need to be increased for reliable results.

## C.5 SAMPLE RETRIEVAL AND WEIGHING

### C.5.1 Impactor Clean-up

The careful disassembly of the impactor and removal of the collected particulate are essential to the success of the test program. The crucial points are to make sure that the collected material stays where it originally impacted and to remove all the particulate. After the sampling run, the impactor should be carefully removed from the duct without jarring it, removed from the probe, and allowed to cool. Disassembly can be difficult in some cases, particularly if the impactor was used at elevated temperatures.

Typically, not all of the particulate which collects in an impactor collects on the substrates. Some accumulates on the interior surfaces, especially in the nozzle. By convention, all of the particles collected upstream of a given impaction stage are assigned to that stage.

The collection of these "misdirected" particles is often troublesome. If the particles are hard and dry, they can be brushed off into the weighing container. A No. 7 Portrait brush or its equivalent is suggested, and care must be taken to prevent brush hairs from contaminating the sample. If the particles are sticky or wet, some type of washdown procedure should be used. The solvent must be considerably more volatile than the particulate matter.

### C.5.2 Drying and Weighing

All of the samples must be dried to constant weight, with 2 hour checks used to establish the uniformity of the weights. Hard, nonvolatile particles are often dried in a convection oven to 100°C, desiccated until cooled to room temperature, weighed, then check weighed. Volatile materials will require some other technique using low temperature. Whatever the technique used, constant weight of the sample with further drying is the criteria to be met.

### C.5.3 Data Logging

Permanent records should be kept of all pertinent information. It is generally necessary to keep records in three places--in the lab with the balance using a bound notebook, and using either looseleaf data forms or a bound notebook at both the inlet and outlet of the control device. Table C2 presents a fairly complete listing of the information required concerning an impactor run. In addition, records of the weighing of the catches must be kept. Notes should be taken on any abnormalities which occur and on the apparent condition of the stage catches.

## C.6 QUALITY ASSURANCE

The field use of cascade impactors is a difficult task. The accuracy required is more appropriate for a laboratory program than for a field test. There are many places in the operational sequence where errors can occur in spite of a conscientious effort to do a good job. Quality assurance attempts to discover inaccuracies before they are propagated throughout the test program. The techniques presented in this section are not the only ways to ensure quality data. However, they have been used successfully in field testing with impactors.

### C.6.1 Impactor Techniques

Glass fiber substrates -- As has been discussed previously, glass fiber substrates are not without problems. Two potentially serious problems are $SO_2$ uptake on the substrate and mechanical or manual abrasion of the filter mat.

## TABLE C2. SAMPLING INFORMATION REQUIRED

Date
Time
Run Code Number
Impactor Type and Identification Number
Operator
Port Number/Sampling Location
Ambient Temperature
Ambient Pressure

Impactor In-Stack or Out-of-Stack
Impactor Orientation
Number of Traverse Points
Stack Pressure
Stack Temperature
Nozzle Diameter/Type
Probe Depth, if used
Stack Pitot Tube Delta P/Stack Gas Velocity

Desired Impactor Flow rate for Isokinetic Sampling
Metering Orifice Identification Number
Metering Orifice Delta P
Impactor Temperature
Scalping Cyclone in Use? Identification
Prefilter Identification
Postfilter Identification
Substrate Set Identification
Pressure Drop Across Impactor

Test Start/End Time; Duration of Test
Gas Meter Start/End Readings? Gas Meter Volume
Agreement Between Meter and Orifice
Volume of Condensible $H_2O$ in Flue Gas
Gas Meter Temperature

The problem of $SO_2$ uptake on the substrate is discussed by Felix _et al._[1] The two approaches available are to use a substrate which does not change weight in the flue gas or to precondition the substrate in filtered flue gas prior to weighing. Using a new glass fiber material, which does not react with the $SO_2$, may alter the particle retention characteristics of the impactor and change the impactor's calibration. This must be checked and the data reported. The use of preconditioned filter mats requires that the glass fiber substrates be preconditioned long enough to reduce the weight change during the expected duration of the impactor runs to 10 percent or less of the minimum stage weight. At the present time, this $SO_2$ reaction phenomenon is not well understood, and only rough guidelines are available. For some common glass fiber materials tested, the saturation times were on the order of 2 to 6 hours at the temperatures tested.

The applicability of the method chosen to overcome this substrate problem must be tested during the presurvey and periodically during the test runs by running blanks.

Glass fiber substrates must be handled carefully to prevent damage and possible loss of fibers. Loose surface fibers should be removed by shaking prior to initial weighing. After weighing, every precaution must be taken to prevent the loss of any part of the substrate. One approach which will quantify the problem of substrate abrasion is to prepare a substrate set, load the impactor, then disassemble and reweigh.

_Greased metal substrates_ -- The problems which occur with the use of greased substrates are usually related to the properties of the grease. A grease which has been applied too heavily or has a low viscosity at operating temperature can be physically blown off the impactor stage. The grease could also react chemically with the flue gas or be excessively volatile at the operating temperature. Again, these phenomenon must be checked during the presurvey and periodically during the test program.

Re-entrainment -- Re-entrainment is the phenomenon of an impacted particle being blown off the stage on which it was collected initially and being recollected downstream. This can be caused by excessive jet velocities or by overloaded stages. The effect of re-entrainment can be serious, because only a few large particles on a small particle stage will considerably affect the size distribution.

One way to spot re-entrainment is to very carefully examine the stage catches. If, for example, a low velocity through the jets resulted in a well-defined pile of particulate and a high velocity sample gave a diffuse deposit, re-entrainment should be suspected at the high sampling rate. Microscopic examination of the lower stages and final filter for large particles (which should have been collected upstream) is another way to check for re-entrainment.

Re-entrainment due to stage overloading can be detected by running two otherwise identical tests for two different test durations. If the two size distributions are not the same, overloading should be suspected at the higher stage loadings.

Impactor leaks -- Two types of leaks can occur with impactors--internal or external. A flow rate versus pressure drop check of a pressure test will pick up most leaks. An internal leak, where part of the airstream is bypassing the proper flow path, will give results similar to re-entrainment. Leak checks must be made at operating temperature.

General procedure -- A general procedure for impactor use, concentrating on quality assurance, has been outlined below:

1. Prepare Impactor
    a. Wash impactor. Use ultrasonic cleaner if available.
    b. Visually check cleanliness. Jets must be clear, sidewalls clean. Must be done in good lighting.
    c. Obtain preweighed substrates and assemble impactor.

2. Sampling
    a. Assemble impactor train and heat to operating temperature.
    b. Leak check the impactor.
    c. Sample with impactor.
    d. Disassemble impactor, examine stage catches and impactor walls. Note any anomalies.

3. Substrate and Re-entrainment Checks
    a. Check during presurvey.
    b. Check substrates if flue gas composition changes significantly.

### C.6.2 Weighing Techniques

Precision and calibration -- The manufacturer's directions should be followed when operating the balance. The balance should be calibrated at least once a day. The repeatability of measurements should be checked by repeatedly weighing a substrate and a test weight.

Technique -- The assembly and disassembly of an impactor should have no effect on the substrate weights. This should be checked by weighing up a set of substrates, assembling them in an impactor, then disassembling and reweighing. Any weight losses from this process should be within the repeatability of the balance (approximately 0.02 milligram for an electrobalance). Dry weight checks are made by desiccating the substrate, weighing, then desiccating again and reweighing. When the agreement is within the repeatability of the balance, dry weight has been achieved.

### C.6.3 General Notes

Spare parts -- The well equipped sampling team will travel with an adequate supply of spares. Improvisation due to an equipment failure can lead to poor quality data.

Flow meters -- At least two flow meters should be used in series. If they do not agree, the problem should be investigated.

Pumps -- Typically, vacuum pumps in sampling trains leak. For this reason, the flow meters should be upstream of the pump.

### C.6.4 Data Analysis

Final filter data -- The fine particulate information obtained from the final filter can sometimes be misleading. It is assumed for analysis that a stage captures everything larger than its $D_{50}$, and captures nothing smaller. A real stage misses some large particles. Under some conditions (including but not limited to re-entrainment), large particles will penetrate to the final filter. In this case the size distribution will be skewed towards the small particles. Microscopic examination of the final filter may provide an indication of this problem. If it occurs, the best choice in data analysis is probably to ignore the final filter on runs where this phenomenon was encountered.

Cumulative size data analysis -- If either a probe or a precutter cyclone is used with an impactor, the resultant probe losses and precutter catches must be included in cumulative size analysis. Failure to do so will lead to an incorrect cumulative distribution.

Inspection of data -- After the data have been collected, they should be examined for any inconsistencies and outliers should be rejected before the final averaging is done.

## C.7 USE OF COMMERCIAL IMPACTORS

### C.7.1 Brink BMS-11 Cascade Impactor

Additional information on the following impactors can be found in Section 2.1.2 under Table 1.

The Brink impactor is a five-stage, low sample rate, cascade impactor, suitable for measurements in high mass loading situations. The Brink uses a single round jet on each of its stages.

Sampling rate -- The usual sampling rates for the Brink are in the range of 9-33 $cm^3/sec$ (0.02 to 0.07 acfm). The sampling rate must be low enough to prevent re-entrainment of particles from the lower stages. With hard, bouncy particulate, the last stage nozzle velocity must be less than 30-35 m/sec with ungreased substrates, and less than 65 m/sec with greased substrates.

Collection substrates and adhesives -- The Brink impactor collection stage is too heavy to use without some type of substrate insert. Foil cups are commonly preformed and fitted into the collection cups of the Brink stages. If grease is to be used, the top stages require about 5 or 6 drops of solution while the bottom stages normally require only about one drop in the center of the cup. Glass fiber substrates cut to fit the collection cups have also been found satisfactory in many situations.

Back up filter -- The Brink back up filter is normally made of binderless glass fiber filter material. Two 2.5 cm diameter disks of filter material are placed under the spring in the last stage of the impactor. The filter is protected by a Teflon O-ring and the second filter disk acts as a support.

Precutter cyclone -- A precutter cyclone for the Brink is not presently commercially available.

Sampling train -- The Brink uses the usual type of sampling train. Orifices on the order of 0.77, 1.52, and 2.29 cm in diameter allow full coverage of its range of sampling rates at reasonable pressure drops.

Brink clean-up -- Careful disassembly of a Brink impactor is necessary for obtaining good stage weights. If a precollector cyclone has been used, all material from the nozzle to the outlet of the cyclone is included with the cyclone catch. All of this material should be brushed onto a small, tared, 2.5 x 2.5 cm aluminum foil square to be saved for weighing. Cleaning the nozzle is also important, especially if it is a small bore nozzle. All material between the cyclone outlet and the second stage nozzle is included with material collected on the first collection substrate. All appropriate walls should be brushed off, as well as around the underside of the nozzle, where as much as 30 percent of the sample has been found.

### C.7.2 Andersen Mark III Stack Sampler

The Andersen impactor is a relatively high sample rate impactor. The normal sample rate is about 236 cm$^3$/sec (0.5 acfm). The Andersen is a multiple jet, round hole impactor.

Sampling rate -- The nominal Andersen sampling rate is given above. As with other cascade impactors, the flow rate must be low enough to prevent re-entrainment of impacted dust.

Collection substrates and adhesives -- Andersen substrates are obtained precut from the manufacturer. The substrates are glass fiber and of two types--one cut for the odd numbered stages, one for the even. As discussed earlier, normal Andersen substrates have a tendency to absorb $SO_2$ on basic sites in the substrate and therefore gain weight.

The Andersen requires careful assembly, as overtightening will cause the substrates to stick to the metal separator rings.

Back up filter -- The Andersen uses a 63.5 mm diameter disk placed above the final F-stage. (This F-stage is an option not normally included with the standard stack head.) The filter should be cut from binderless glass fiber filter material, such as Reeve-Angel 934AH filter paper.

Precutter cyclone -- A precutter cyclone for the Andersen is available from the manufacturer. It is necessary to have a 6-inch diameter or larger sampling port when using the precutter cyclone with its nozzle.

Andersen sampling train -- The Andersen requires the usual type of sample train. The pumping and metering systems of the commercial EPA Method 5 mass sampling trains are appropriately sized for use with the Andersen.

Care should be exercised never to allow a gas flow reversal to occur through the impactor. Material could be blown off the collection substrate onto the underside of the jet plate or the collection substrates could be disturbed. A check valve or maintenance of a very low flow while removing the impactor from the duct avoids this problem.

Andersen clean-up -- Cleaning an Andersen impactor is difficult. Foils should be cut to hold the substrates, and each foil and substrate weighed together before and after the run. For disassembly, the foil to hold the stage 1 substrate should be laid out. Next the nozzle and entrance cone should be brushed out and onto the foil. Then the material on stage 0 should be brushed onto the foil. The stage 1 filter substrate material should then be placed on the foil and, finally, the top of the stage 1 plate, O-ring, and cross piece should be brushed off. Depending on how tightly the impactor was assembled, some filter material may stick to the O-ring edge contacting the substrate. This should be carefully brushed onto the appropriate foil. This process is continued through the lower stages. Finally, the after filter is carefully removed.

### C.7.3 University of Washington Mark III (Pilat) Impactor

The Mark III impactor is a seven-stage, high flow rate device with generally the same characteristics as the Andersen. The Mark III is a round hole, multiple jet imapctor.

*Sample rate* -- The Mark III sampling rate is on the order of 236 $cm^3/sec$ (0.5 scfm). The flow rate must be low enough to keep scouring of impacted particles to a minimum.

*Collection substrates and adhesives* -- The Mark III has often been used with supplementary foil (aluminum or stainless steel) substrates. These substrates require the use of grease for easily re-entrained particles. Enough of the grease solution is placed evenly on the substrate to adequately cover the area under the jets. The normal cautions on the use of greased substrates apply as discussed in the text.

*Precutter cyclone* -- A BCURA (British Coal Utilization Research Association) designed precutter cyclone is available from the manufacturer.

*Mark III sampling train* -- As the Mark III is a high flow rate device, its sampling train is similar to that of the Andersen.

*Mark III clean-up* -- Mark III impactor clean-up is similar to that for the Brink. Some problems have been noted with O-rings sticking rather tenaciously and care must be exercised not to dislodge the sample while trying to separate the stages.

### C.7.4 Meteorology Research, Inc. (MRI) Model 1502 Cascade Impactor

The MRI impactor is a high flow rate sampler. The body of the instrument is constructed from quick-disconnect rings which allow flexibility in configuration of the impactor and a positive gas seal between stages. The impactor uses multiple round jets in its stages.

*Sampling rate* -- The sampling rate is nominally 236 $cm^3/sec$ (0.5 acfm) in the seven-stage configuration. Higher flow rates have been used by removing the last stage.

*Collection substrates and adhesives* -- The MRI collection disc is a self-supporting foil (316 stainless steel) which is functionally similar to the collection cup or tray and inserts used in other impactors. The collection discs are mass produced and normally are used only once and discarded.

Grease applied as described earlier is recommended for most applications.

Back-up filter -- The MRI impactor has a built-in filter holder for 47 mm diameter filters. Normally, binderless glass filters are used. Filter losses can be prevented by placing tared Teflon washers on both sides of the filter during the test.

MRI sampling train -- The MRI sampling train is similar to that of the Andersen.

MRI clean-up -- The clean up of the MRI impactor is similar to the Brink. The device is clamped in a vice and all of the sections and nozzles are loosened with wrenches. The wall losses are carefully brushed onto the appropriate collection disc. Care is taken not to brush contamination from the threads into the sample. A tared foil dish is used to hold the back up filter. Any worn O-rings should be replaced and the whole unit carefully cleaned before the next test.

### C.7.5 Sierra Model 226 Source Cascade Impactor

The Sierra impactor is a six-stage, high sample rate cascade impactor. The Sierra instrument uses a radial-slot design.

Sampling rate -- The Sierra impactor has a nominal sampling rate 236 $\text{cm}^3$/sec (0.5 acfm). The flow rate must be low enough to prevent re-entrainment of particles.

Collection substrates and adhesives -- Substrates for the Sierra are obtained precut from the manufacturer. These are glass fiber substrates and should be checked for weight gain. Stainless steel substrates are also available and these should normally be coated with grease as described earlier.

Back-up filter -- The back-up filter uses a 47 mm glass fiber filter mat. It is supported by a screen from below.

Precutter cyclone -- A precutter cyclone is available from the manufacturer.

Sampling train -- The sampling train for the Sierra is similar to that of the Andersen.

Clean-up -- Clean-up of the Sierra is fairly similar to Andersen clean-up. Care should be taken to be sure the glass fiber substrates are removed intact.

## C.8 DATA ANALYSIS

### C.8.1 General Discussion

The majority of impactor data reduction is done using a technique referred to as the "$D_{50}$" method. In this method it is assumed that all of the particles caught by an impactor stage consist of particles having diameters equal to or greater than the $D_{50}$ of that stage, but less than the diameter of the stage above. The mass caught by the first stage or cyclone is considered to be equal to or greater than the $D_{50}$ of that stage, but smaller than the largest particle diameter present in the aerosol. The largest diameter may be determined, approximately by microscopic examination of the stage catch, or it may be assigned some reasonable arbitrary value, say 100 μm.

Particle size distributions may be presented on a differential or a cumulative basis. When using the $D_{50}$ method, either type of presentation may be easily employed.

The size parameter reported can be aerodynamic diameter, aerodynamic impaction diameter, or Stokes diameter. In all cases, the particles are assumed to be spherical. The method of reporting diameters depends to a large extent upon the ultimate use of the size distribution information. For this reason it is suggested that the data be reported in three parallel sets: one set based on aerodynamic impaction diameter, one based on aerodynamic diameter, and one based on the Stokes diameter.

### C.8.2 Calculation of Impactor Stage $D_{50}$'s

The reduction of field data obtained with a cascade impactor can sometimes be troublesome and time consuming because of the computations involved. The equations below are based on the motion of particles in the Stokes regime for which the Reynolds number is less than 1000. Although this is not always true for impactors, the equations are often a good approximation. The basic equation that defines the theoretical impaction behavior of a given stage of a cascade impactor is

$$\psi = \frac{\rho_p D^2 V_j C}{18 \mu D_j} , \qquad \text{(C7)}$$

where $\psi$ is the Stokes number,

$\rho_p$ is the particle density, $gm/cm^3$,

$V_j$ is the velocity of the gas (and particles) in the impactor jet (cm/sec),

$\mu$ is the gas viscosity, poise,

$D_j$ is the diameter (width for slots) of the jet, cm, and

$C$ is the slip correction factor.

If the value of $\psi$ for 50 percent collection, $\psi_{50}$, can be determined, equation C7 can be inverted to give the stage $D_{50}$ for a wide range of test conditions. Historically, the experimental values reported by Ranz and Wong[7] have been used. These are:

For round jets, $\psi_{50} = 0.145$, and
For rectangular jets, $\psi_{50} = 0.44$.

Subsequent studies, however, have shown that there is no universal value for $\psi_{50}$ and the actual value must be determined by calibration for each impactor design. Several papers and reports are available which tabulate stage constants of different impactors, and outline procedures for impactor calibration.[illegible]

From equation C7,

$$D_{50} = \left(\frac{18\psi_{50}\, D_j}{C\rho_p V_j}\right)^{\frac{1}{2}} \qquad \text{(C8)}$$

As equation (C8) is written, with the actual particle density and the calculated slip correction factor, it defines the Stokes diameter. If the particles are treated as if their density, $\rho_p$, was 1.0, equation (C8) defines the aerodynamic diameter. If the slip correction factor is also assumed to be equal to 1.0, the aerodynamic impaction diameter is defined by equation (C8).

Since C, the slip correction factor, contains D, this equation must be solved by iteration where $D_{50}$ and C are calculated alternately.

Equation C8 may be written more conveniently in terms of the test parameters: For round jet impactors

$$D_{50} = \left(\frac{14.1\ \psi_{50}\mu D_j^3 X_j P_j}{C\rho_p Q_s P_s}\right)^{1/2}, \tag{C9}$$

where $X_j$ is the number of jets on the stage,

$P_j$ is the absolute pressure downstream of the jet(s), mm Hg,

$P_s$ is the absolute pressure in the stack, mm Hg, and

$Q_s$ is the sample flow rate, cm³/sec.

and for rectangular jet impactors

$$D_{50} = \left(\frac{18\ \psi_{50}\mu W_j^2 L_j X_j}{C\rho_p Q_s P_s}\right)^{1/2} \tag{C10}$$

where $W_j$ is the jet width, cm, and

$L_j$ is the total jet length, cm.

One approach that can be used to simplify the computations is to develop curves for the impactor stage cut points at one set of conditions--e.g., air at standard conditions and a particle density of 1.0. Then a suitable correction factor can be applied to these curves for the actual sampling conditions. Unfortunately, further steps are involved in making the correction factor simple enough to be of value. Therefore, the use of this type of approach suffers from some restrictions.

All of the assumptions and calculations involved in going from equations C9 to C10 to the calibration curve can be quite awkward, particularly in cases where different types of sources are being sampled. The best and easiest approach is to write or obtain a computer program based on the rigorous equations given initially. Such a program can calculate impactor stage cut points, compute concentrations of particles in each size range, determine the precipitator efficiency, and plot graphs.

A sophisticated computer data reduction program is available from the EPA and also less powerful programs are available for the Hewlett Packard HP-65 and HP-25 programmable calculators.[114]

### C.8.3 Cumulative Particle Size Distributions

Impactor data may be presented on a cumulative basis by summing the mass on all the collection stages and back up filter, and plotting the fraction of the mass below a given size versus size. This is frequently done on special log-probability paper. Semi-log paper may be preferable for distributions that are not log-normal.

Cumulative distributions are very easy to understand and present the data with clarity. For this reason, they should be presented as part of each particle sizing report. Cumulative distributions do have a couple of disadvantages when compared to differential distributions. An error in stage weight will be propagated throughout a cumulative analysis, but will be isolated by the differential approach. Also the differential method does not involve the use of total mass concentration or total size distribution from diameters of zero to inifinity, and so is useful in comparing instruments with overlapping but different size fractionation ranges and different stage cut points while cumulative analysis is not.

When cumulative plots are used, the abscissa is normally the logarithm of the particle diameter and the ordinate is the weight percent smaller than this size. The value of the ordinate at a given $(D_{50})_k$ would be

$$\text{Weight percent smaller than } (D_{50})_k = \frac{\sum_{i=o}^{k-1} \Delta M_i}{\sum_{i=o}^{N} \Delta M_i} \times 100\%. \quad \text{(C11)}$$

where $i = o$ corresponds to the filter,
$i = k$ corresponds to the stage under study, and
$i = N$ corresponds to the coarsest jet or cyclone.

This equation requires that the stages be counted from the final filter up. There is no $(D_{50})_o$, as the "o" corresponds to the filter. $(D_{50})_1$ is the cut point of the last stages, which collects mass, $\Delta M_1$.

### C.8.4 Differential Size Distributions

Differential particle size distributions are used to plot the relative concentration versus particle diameter. The area under the frequency curve, between two designated diameters, is equal to the mass of particles in that size range. Differential curves may be obtained directly from the reduced impactor data, or by differentiation of curves fitted to the cumulative particle size distributions.

Many cascade impactors are designed so that the relationship between successive stage $D_{50}$'s is logarithmic. For this reason, and to minimize graphical scaling problems, the differential particle size distributoins are plotted on log-log or semi-log paper with $\Delta M/\Delta(\log D)$ as the ordinate and geometric mean of $\Delta(\log D)$ as the abscissa. The mass on stage "n" is designated by $M_n$ and is, in approximation, the particulate mass with diameters between $(D_{50})_n$ and $(D_{50})_{n+1}$. The $\Delta(\log D)$ associated with $\Delta M_n$ is $\log(D_{50})_{n+1} - \log(D_{50})_n$. Using these approximations, the derivative term (ordinate) associated with stage "n" is:

$$\Delta M/\Delta(\log D)\big|_n = \frac{\Delta M_n}{\Delta(\log D_{50})\big|_n} = \frac{\text{Mass on Stage "n"}}{\log(D_{50})_{n+1} - \log(D_{50})_n}, \tag{C12}$$

and the abscissa, $D_{geo}$, is

$$\left[(D_{50})_{n+1} \times (D_{50})_n\right]^{\frac{1}{2}}. \tag{C13}$$

Usually, smooth curves are fitted through these computed points for convenience in averaging the data and calculating the precipitator efficiency. As many tests as possible should be obtained for each precipitator operating conditions and the results averaged after outliers have been identified and discarded.

The fractional penetration of the precipitator is determined by dividing the magnitude of the outlet $\Delta M/\Delta(\log D)$ plot by the magnitude of the inlet at several corresponding values of $D_{geo}$. The efficiency is equal to the penetration subtracted from unity, multiplied by 100%. Both the efficiency and penetration can be conveniently plotted on a single log-probability graph.

### C.8.5 Confidence Limits of Reduced Data

After data from the individual cascade impactor runs have been reduced, then averages for these inlet and outlet tests can be calculated. Generally the data are presented as 1) Cumulative Mass Versus Particle Size, 2) Cumulative Per Cent Mass Versus Particle Size, and 3) ΔM/Δ(logD) Versus Geometric Mean Particle Size. To calculate the Upper and Lower 90% Confidence Limits for the data, the following equations are used:

$$UCL_{90} = \text{Average} + \frac{t_{90}\sigma}{\sqrt{N}} = \text{Average} + C.I._{90} \qquad (C14)$$

$$LCL_{90} = \text{Average} - \frac{t_{90}\sigma}{\sqrt{N}} = \text{Average} - C.I._{90} \qquad (C15)$$

where

Average = The average of the N values for ΔM/ΔlogD or ΔN/ΔlogD at a particular particle size.

$t_{90}$ = The Student's t distribution for 90% confidence limits with N sets of data.

$\sigma$ = The Standard Deviation of the N sets of data.

N = The number of data values in the average.

The average cumulative mass and cumulative per cent graphs are obtained from ΔM/ΔlogD data by piecewise integration, after discarding outliers. The confidence interval of the cumulative graph, at a particular size, is equal to the square root of the sum of the squares of the confidence intervals of each size increment of the differential graph less than or equal to that size.

Clearly, the average values are more reliable and the confidence limits are small when the number of data points is large.

A determination of precipitator fractional penetration is made by taking the ratio of the average ΔM/ΔlogD outlet values to the average ΔM/ΔlogD inlet values at a series of particle sizes. The confidence limits of the calculated penetration are given by:

$$UCL_{95} = \text{Average Penetration} + CIP_{95} = P + CIP_{95} \quad (C16)$$

and

$$LCL_{95} = \text{Average Penetration} - CIP_{95} = P - CIP_{95} \quad (C17)$$

where:

$$CIP_{95} = \left[\frac{(C.I._{95},\ \text{Outlet})^2}{(\text{Inlet Average})^2} + P^2\frac{(C.I._{95},\ \text{Inlet})^2}{(\text{Inlet Average})^2}\right]^{1/2} \quad (C18)$$

and

$$P = \frac{\text{Outlet Average}}{\text{Inlet Average}} .$$

## C.8.6 CASCADE IMPACTOR DATA REDUCTION--SAMPLE CALCULATION

This section contains a detailed description of the calculations that are required to derive particle size distributions and a precipitator fractional efficiency curve from raw impactor data. The specific example given is for a single hypothetical test performed with an Andersen impactor. The calculation procedures outlined here can be used with any impactor, however. Normally the results of all tests made under the same process and precipitator operating conditions are grouped and averaged, and confidence limits calculated as described in Section C.8.5.

This discussion is based on Table C3 which was generated by a computer program. All of the calculations, however, can be done on programmable calculators. In the example shown, the data is reduced using a particle density of 1.35 gm/cm$^3$; thus the diameters reported are Stokes diameters.

## TABLE C3

## SAMPLE CALCULATION - INPUT DATA AND RESULTS

Hypothetical Andersen

Impactor Flowrate = 0.500 ACFM        Impactor Temperature = 400.0 F = 204.4 C        Sampling Duration = 20.00 Min

Impactor Pressure Drop = 1.5 In. of Hg        Stack Temperature = 400.0 F = 204.4 C

Assumed Particle Density = 1.35 gm/cu.cm.        Stack Pressure = 26.50 In. of Hg        Max. Particle Diameter = 100.0 Micrometers

Gas Composition (Percent)        CO2 = 0.00        CO = 0.00        N2 = 78.50        O2 = 20.50        H2O = 1.00

Calc. Mass Loading = 8.0712E-03 gr/acf        1.4946E-02 gr/dncf        1.8470E+01 mg/acm        3.4207E+01 mg/dncm

| Impactor Stage | S1 | S2 | S3 | S4 | S5 | S6 | S7 | S8 | FILTER |
|---|---|---|---|---|---|---|---|---|---|
| Stage Index Number | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
| D50 (Micrometers) | 10.74 | 9.85 | 6.45 | 4.16 | 2.22 | 1.29 | 0.69 | 0.33 | |
| Mass (Milligrams) | 0.72 | 0.40 | 0.53 | 0.09 | 0.38 | 1.43 | 1.25 | 0.04 | 0.39 |
| MG/DSCM/STAGE | 4.71E+00 | 2.62E+00 | 3.47E+00 | 5.89E-01 | 2.48E+00 | 9.37E+00 | 8.19E+00 | 2.62E-01 | 2.55E+00 |
| Cum. Percent Of Mass Smaller Than D50 | 86.24 | 78.59 | 68.46 | 66.74 | 59.47 | 32.13 | 8.27 | 7.46 | |
| Cum. (MG/ACM) Smaller Than D50 | 1.59E+01 | 1.45E+01 | 1.26E+01 | 1.23E+01 | 1.10E+01 | 5.93E+00 | 1.53E+00 | 1.38E+00 | |
| Cum. (GR/ACF) Smaller Than D50 | 6.96E-03 | 6.34E-03 | 5.53E-03 | 5.39E-03 | 4.80E-03 | 2.59E-03 | 6.64E-04 | 6.02E-04 | |
| Cum. (GR/DSCF) Smaller Than D50 | 1.29E-02 | 1.17E-02 | 1.02E-02 | 9.98E-03 | 8.89E-03 | 4.80E-03 | 1.24E-03 | 1.12E-03 | |
| Geo. Mean Dia. (Micrometers) | 3.28E+01 | 1.03E+01 | 7.96E+00 | 5.17E+00 | 3.05E+00 | 1.69E+00 | 9.41E-01 | 4.74E-01 | 2.33E-01 |
| DM/DLOGD (MG/DSCM) | 4.86E+00 | 7.34E+01 | 1.79E+01 | 3.25E+00 | 8.99E+00 | 3.99E+01 | 2.98E+01 | 8.30E-01 | 8.47E+00 |
| DN/DLOGD (NO. PARTICLES/DSCM) | 1.95E+05 | 1.02E+08 | 5.03E+07 | 3.33E+07 | 4.48E+08 | 1.76E+10 | 5.03E+10 | 1.26E+10 | 9.76E+11 |

Normal or standard conditions are 21°C and 760 mm Hg

For aerodynamic, or aerodynamic impaction diameters, $\rho$ or $\rho$ and $C\rho$, are set to unity, respectively. ($\rho$ is particle density and $C$ is the slip correction factor.)

Information obtained from the data log sheets for each test is printed at the top of Table C1. The maximum particle diameter is measured by examining the particles collected on the first stage (or first cyclone) with an optical microscope. Gas analysis samples are taken at the same time the impactor is run. The mass loading is calculated from the total mass of the particles collected by the impactor, and listed in four different systems of units after the heading CALC. MASS LOADING. The symbols are defined as:

GR/ACF - grains per actual cubic foot of gas at stack conditions of temperature, pressure, and water content.

GR/DSCF - grains per dry standard cubic foot of gas at engineering standard conditions of the gas. Engineering standard conditions are defined as 0% water content, 70°F, and 29.92 inches of Hg.

MG/ACM - milligrams per actual cubic meter of gas at stack conditions of temperature, pressure, and water content.

MG/DSCM - milligrams per dry standard cubic meter of gas at engineering standard conditions of the gas. Engineering standard conditions are defined as 0% water content, 21°C and 760 mm of Hg (Torr).

The conditions at which the impactor was run to determine stage $D_{50}$ cut points. These are calculated by iterative solution of the following equations:

$$D_{50_i} = \left[ \frac{14.1\ \mu D_{c_i}{}^3 \psi_{50_i} P_{s_i} N_i}{\rho_p\ Q\ P_0\ C_i} \right]^{\frac{1}{2}} \tag{C19}$$

$$C_i = 1 + \frac{2\ \lambda_i}{D_{50_i} \times 10^{-4}} \left[ 1.23 + 0.41\ \mathrm{EXP}\left(-0.44 \frac{D_{50_i}}{\lambda_i} \times 10^{-4}\right)\right] \tag{C20}$$

where $D_{50_i}$ = stage (i) cut point (cm),

$\mu$ = gas viscosity (poise),

$D_{c_i}$ = stage jet diameter (cm),

$P_{s_i}$ = local pressure at stage jet (mm Hg),

$\rho_p$ = particle density (gm/cm$^3$),

$Q$ = impactor flow rate (cm$^3$/sec),

$P_0$ = ambient pressure at impactor inlet (mm Hg),

$C_i$ = Slip Correction Factor,

$\lambda_i$ = gas mean free path (cm),

$N_i$ = number of holes per stage (i), and

$\psi_{50_i}$ = Stokes number of stage (i).

To find the pressure $P_{s_i}$ at each impactor stage, the following equation is used:

$$P_{s_i} = P_0 - (F_i)(DP), \tag{C21}$$

where $P_0$ is the ambient pressure at the impactor inlet,

$F_i$ is the fraction of the total impactor pressure drop at each stage, and

DP is the total pressure drop across the impactor. The total pressure drop DP across the impactor is given by the following equation.

$$DP = K\,Q^2\,\rho\,MM, \tag{C22}$$

where K = Empirically determined constant for each impactor,

Q = Flow rate through impactor ($cm^3/sec$),

= Gas density ($gm/cm^3$), and

MM = Mean Molecular Weight of gas (gm/gm-mole).

To calculate the gas mean free path, $\lambda_i$, for each impactor stage, the following equation is used:

$$\lambda_i = \frac{2\mu}{1.013 \times 10^6\,P_{s_i}} \times \sqrt{\frac{8.31 \times 10^7\,T_K}{3\,MM}} \tag{C23}$$

where $\mu$ is the gas viscosity (poise),

$P_{s_i}$ is the pressure at each impactor stage (atm),

$T_K$ is the gas temperature at the impactor stage (°K), and

MM is the average flue gas molecular weight.

To find the viscosity of the flue gas, $\mu$, the viscosity of the pure gas components of the flue gas must first be found. Viscosity is a function of temperature, and the temperature difference in different flue gases can be quite significant. The following equations (derived from curves fitted to viscosity data from the Handbook of Chemistry and

Physics, Chemical Rubber Company Publisher, 54 Edition, 1973-1974, pp. F52-55), are used to find the viscosities of $CO_2(\mu_1)$, $CO(\mu_2)$, $N_2(\mu_3)$, $O_2(\mu_4)$ and $H_2O(\mu_5)$.

$\mu_1 = 138.494 + 0.499T - 0.267 \times 10^{-3}T^2 + 0.972 \times 10^{-7}T^3$
$\mu_2 = 165.763 + 0.442T - 0.213 \times 10^{-3}T^2$
$\mu_3 = 167.086 + 0.417T + 0.417 - 0.139 \times 10^{-3}T^2$
$\mu_4 = 190.187 + 0.558T - 0.336 \times 10^{-3}T^2 + 0.139 \times 10^{-6}T^3$
$\mu_5 = \ 87.900 + 0.374T + 0.238 \times 10^{-3}T^2$

where T is the temperature of the flue gas in degrees Celsius. The units of $\mu$ are $10^{-6}$ g/cm-sec. Next, these values of $\mu_1$ through $\mu_5$ are used in a general viscosity equation for a mixture of any number of components (See "A Viscosity Equation for Gas Mixtures" by C. R. Wilke, Journal of Chemical Physics, Volume 8, Number 4, April 1950, page 517) used to find the viscosity of the flue gas:

$$\mu = \sum_{i=1}^{n} \frac{\mu_i}{\left[1 + \frac{1}{X_i} \sum_{\substack{j=1 \\ j \neq i}}^{j=n} X_j \phi_{ij}\right]}$$

where $\phi_{ij}$ is given by the equation:

$$\phi_{ij} = \frac{\left[1 + (\mu_i/\mu_j)^{1/2}(M_j/M_i)^{1/4}\right]^2}{(4/\sqrt{2})\left[1 + (M_i/M_j)\right]^{1/2}} , \qquad \text{(C25)}$$

and M = molecular weight of a component in the mixture,
X = mole fraction of a component in the misutre,

$\mu$ = viscosity, gm/cm-sec; $\mu_1$, $\mu_2$, etc. refer to the pure components at the temperature and pressure of the mixture, $\mu$ is the viscosity of the mixture, and

$\phi$ = dimensionless constant defined above.

Below these data the information pertinent to each stage is summarized in columnar form in order to decreasing particle size from left to right. Thus S1 is the first stage, S8 is the last stage, and FILTER is the back-up filter. If a cyclone was used, then to the left of S1 a column labelled CYC would appear and information relevant to the cyclone would be listed in this column. Beneath each impactor stage number is listed the corresponding stage index numbers, which also serve as identification for the stages. Directly beneath these listings is the stage cut point calculated from Equations C19 and C20 for the actual test conditions. It is labelled $D_{50}$ and is given in micrometer units. The stage weights are likewise listed for the respective stages, labelled W/SS and are in milligram units.

The mass loadings per unit volume of gas sampled indicated by the stage weights are labelled MG/DSCM/STAGE and are written in milligrams per dry standard cubic meter. The /STAGE indicates that it is not a cumulative. It is calculated for a particular stage j by the formula

$$\text{MG/DSCM/STAGE}_j = \frac{\text{MASS}_j}{\text{SAMPLING DURATION (minutes)}}$$

$$\times \frac{\text{35.31 cubic feet/cubic meter}}{\text{FLOWRATE (ACFM)}} \times \frac{\text{Absolute Stack Temperature}}{\text{Absolute Standard Temperature}}$$

$$\times \frac{\text{Absolute Standard Pressure}}{\text{Absolute Stack Pressure}} \times \frac{1}{(1 - \text{Fraction of } H_2O)} \qquad \text{(C26)}$$

where absolute means the temperature and pressure are in absolute units-degrees Rankin or degrees Kelvin for temperature, and atmosphere, inches or millimeters of mercury for pressure. For S1,

$$\text{MG/DSCM/STAGE}_1 = \frac{.72\ \text{mg}}{20\ \text{min}} \times \frac{35.31\ \text{cubic feet/cubic meter}}{0.500\ \text{ACFM}}$$

$$\times \frac{(400 + 460)°\text{R}}{(70 + 460)°\text{R}} \times \frac{29.92\ \text{in. Hg}}{26.50\ \text{in. Hg}} \times \frac{1}{(1.0 - 0.01)} = 4.71\ \text{mg/DSCM}$$

The subscripts indicate stage index numbers.

The percent of the mass of particles with diameters smaller than the corresponding $D_{5j}$ is called the **CUMULATIVE PERCENT OF MASS SMALLER THAN $D_{5j}$**. It is the cumulative mass at stage j divided by the total mass collected on all the stages, and converted to a percentage:

$$\text{CUM }\%_j = \frac{\sum_{i=j+1}^{9} \text{MASS}_i}{\text{Total Mass}} \times 100 \qquad \text{(C27)}$$

For example, for S6, the cumulative percent is given by

$$\text{CUM }\%_6 = \frac{\text{MASS}_7 + \text{MASS}_8 + \text{MASS}_9}{\text{Total Mass}} \times 100$$

$$= \frac{1.25\ \text{mg} + 0.04\ \text{mg} + 0.39\ \text{mg}}{5.24\ \text{mg}} \times 100 = 32.06\%$$

For S8, the mass of the particulate collected on the filter is used,

$$\text{CUM }\%_8 = \frac{\text{MASS}_9}{\text{Total Mass}} \times 100 \qquad \text{(C28)}$$

$$= \frac{0.39\ \text{mg}}{5.24\ \text{mg}} \times 100$$

$$= 7.44\%$$

Note that the apparent error in the least significant figures of the calculated percentages is due to using masses from the computer printout which have been rounded off to two decimal places before printing.

The cumulative mass loading of particles smaller in diameter than the corresponding $D_{j}$, in milligrams per actual cubic meter (CUM. (MG/ACM) SMALLER THAN $D_{j}$) for a particular stage j is given by the formula

$$\text{CUM.(MG/ACM)}_j = \frac{\sum_{i=j+1}^{9} \text{MASS}_i}{\text{sampling duration (min)}} \times \frac{\text{35.31 cubic feet/cubic meter}}{\text{FLOWRATE (ACFM)}} \qquad \text{(C29)}$$

From the information at the top of the computer print-out sheet, the flow rate is 0.500 actual cubic feet per minute (ACFM) and the sampling duration is 20.00 minutes. Therefore, for S4,

$$\text{CUM.(MG/ACM)}_4 = \frac{\text{MASS}_5 + \text{MASS}_6 + \text{MASS}_7 + \text{MASS}_8 + \text{MASS}_9}{\text{20 minutes}}$$

$$\times \frac{\text{35.31 cubic feet/ cubic meter}}{\text{0.500 ACFM}} = \text{12.3 mg/ACM}$$

For S8, the mass of the particulate collected on the filter is again used,

$$\text{CUM.(MG/ACM)}_8 = \frac{\text{MASS}_9}{\text{20 minutes}} \times \frac{\text{35.31 cubic feet/cubic meter}}{\text{0.500 ACFM}}$$

$$= \frac{\text{0.39 mg}}{\text{20 minutes}} \times \frac{\text{35.31 cubic feet/cubic meter}}{\text{0.500 ACFM}}$$

$$= \text{1.38 mg/ACM}$$

The cumulative mass loading of particles smaller in diameter than the corresponding $D_{50}$ in grains per actual cubic foot (CUM. (GR/ACF) SMALLER THAN $D_{50}$) for a particular stage j is given by the formula

$$\text{CUM.(GR/ACF)}_j = \frac{\text{CUM.(MG/ACM)}_j}{2.288\ \frac{\text{grams/cubic meter}}{\text{grains/cubic foot}} \times 1000\ \text{mg/gram}} \tag{C30}$$

For S7,

$$\text{CUM.(GR/ACF)}_7 = \frac{1.52\ \text{mg/ACM}}{2.288\ \frac{\text{grams/cubic meter}}{\text{grains/cubic foot}} \times 1000\ \text{mg/gram}}$$

$$= 6.64 \times 10^{-4}\ \text{grains/ACF}$$

The cumulative mass loading of particles smaller in diameter than the corresponding $D_{50}$ in grains per dry standard cubic foot (CUM. (GR/DSCF) SMALLER THAN $D_{50}$) is calculated to show what the above cumulative would be for one cubic foot of dry gas at 70°F and at a pressure of 29.92 inches of mercury. For a particular stage j,

$$\text{CUM.(GR/DSCF)}_j = \text{CUM.(GR/ACF)}_j$$

$$\times \frac{\text{Absolute Stack Temperature}}{\text{Absolute Standard Temperature}} \times \frac{\text{Absolute Standard Pressure}}{\text{Absolute Stack Pressure}}$$

$$\times \frac{1}{(1-\text{Fraction of } H_2O)} \tag{C31}$$

where absolute means the temperature and pressure are in absolute units-degrees Rankin or degrees Kelvin for temperature, and atmospheres, inches or millimeters of mercury for pressure. For S1,

$$\text{CUM. (GR/DSCF)}_1 = 6.96 \times 10^{-3}\ \text{gr/ACF}$$

$$\times \frac{(400 + 460)\,^\circ R}{(70 + 460)\,^\circ R} \times \frac{29.92\ \text{in. Hg}}{26.50\ \text{in. Hg}} \times \frac{1}{(1.00-0.01)} = 1.29 \times 10^{-2}\ \text{gr/DSCF}$$

The particle size distribution may be presented on a differential basis which is the slope of the cumulative curve.

Differential size distributions may be derived two ways:

1. Curves may be fitted to the graphs of cumulative mass vs. particle size, and then the differential curves (slope) of each test would be calculated by taking finite differences along the ordinate and abscissa of the fitted curve.

2. The finite differences may be taken equal to the differences in $D_{50}$'s from stage to stage (abscissa) and the particulate mass on each stage (ordinate). This technique was used to calculate the differential size distribution data in Table C3, and is described in detail in the following paragraphs.

If we define the terms:

$$\Delta M_j = \text{MG/DSCM/STAGE}_j \text{ and}$$

$$(\Delta \log D)_j = \log_{10}(D_{50_{j-1}}) - \log_{10}(D_{50_j}), \text{ then}$$

$$\left(\frac{\Delta M}{\Delta \log D}\right)_j = \frac{\text{MG/DSCM/STAGE}_j}{\log_{10}(D_{50_{j-1}}) - \log_{10}(D_{50_j})} \qquad \text{(C32)}$$

Because the computer printer does not contain Greek letters, the computer print-out sheet reads DM/DLOGD instead of ΔM/ΔLOGD. For S6,

$$\left(\frac{\Delta M}{\Delta LOGD}\right)_5 = \frac{9.35\ \text{mg/DSCM}}{\log_{10}(2.22) - \log_{10}(1.29)} = 39.7\ \text{mg/DSCM}$$

Note that ΔM/ΔLOGD has the dimensions of the numerator since the denominator is dimensionless. In the calculation for S1, a maximum particle diameter is used. For this example, MAX. PARTICLE DIAMETER = 100.0 micrometers.

$$\left(\frac{\Delta M}{\Delta LOGD}\right)_1 = \frac{4.71\ \text{mg/DSCM}}{\log_{10}(100) - \log_{10}(10.74)} = 4.86\ \text{mg/DSCM}$$

For the filter stage, the $D_{50}$ is arbitrarily chosen to be one-half of the $D_{50}$ for stage eight (S8). For this example, it is chosen to be 0.33 micrometers/2 = 0.165 micrometers. Thus,

$$\left(\frac{\Delta M}{\Delta LOGD}\right)_F = \frac{2.55\ \text{mg/DSCM}}{\log_{10}(0.33) - \log_{10}(0.165)} = 8.47\ \text{mg/DSCM}$$

The geometric mean diameter in micrometers (GEO. MEAN DIA. (MICROMETERS)) for a particular stage j is given by the formula

$$\text{GEO. MEAN DIA.}_j = \sqrt{D_{50_j} \times D_{50_{j-1}}} \qquad (C33)$$

For S8,

$$\text{GEO. MEAN DIA.}_8 = \sqrt{0.33 \times 0.69}\ \text{micrometers}$$
$$= 0.477\ \text{micrometers}$$

As in the ΔLOGD calculation, we again use the maximum particle diameter for the stage one calculation and one-half the $D_{50}$ for stage eight for the filter stage calculation.

For S1,

$$\text{GEO. MEAN DIA.}_1 = \sqrt{10.74 \times 100.0} \text{ micrometers}$$
$$= 32.8 \text{ micrometers}$$

For the filter,

$$\text{GEO. MEAN DIA.}_F = \sqrt{0.165 \times 0.33} \text{ micrometers}$$
$$= 0.23 \text{ micrometers}$$

A differential number distribution (for comparison with ultrafine data) can also be derived. Since $\Delta M_j$ = MG/DSCM/STAGE$_j$ is the mass per unit volume for stage j then we can define $\Delta N_j$ as $\Delta N_j$ = NUMBER OF PARTICLES/DSCM/STAGE$_j$ or the number of particles per unit volume for stage j. Now $\Delta M_j$ and $\Delta N_j$ are related by the equation $M_j = N_j \times m_p$, where $m_p$ is the average mass of the particles collected on one stage. Dividing both sides of the equation by $m_p \times \Delta$LOGD yields

$$\frac{(\Delta M/\Delta \text{LOGD})_j}{m_p} = \left(\frac{\Delta N}{\Delta \text{LOGD}}\right)_j \qquad \text{(C34)}$$

Now $m_p = \rho_p V_p$ where $\ _p$ is the assumed particle density and $V_p$ is the average volume of one particle on one stage. To obtain $m_p$ in milligram units when $\rho_p$ is in grams per cubic centimeter and $V_p$ is in cubic micrometers, certain conversion factors must be used. The complete formula, using the correct conversion factors and the expression $(4/3)(\pi)(D/2)^3$ for $V_p$ where d is the geometric mean diameter in micrometers, is:

$$m_p = \rho_p\left(\frac{10^3\ \text{mg}}{1\ \text{gm}}\right)\left(\frac{4\pi}{3}\right)\left(\frac{d}{2}\right)^3\left(\frac{10^{-12}\ \text{cm}^3}{1\ \text{cubic micrometer}}\right)$$

$$= (5.23599 \times 10^{-10})\rho_p d^3.$$

Therefore,

$$\left(\frac{\Delta N}{\Delta LOGD}\right)_j = \frac{(\Delta M/\Delta LOGD)_j}{5.23599 \times 10^{-10}\rho_p d^3}$$

where ΔM/ΔLOGD is in units of mg/DSCM, $\rho_p$ is in gm/cm$^3$, d is in microns, and ΔN/ΔLOGD is in number of particles/DSCM. For S3,

$$\left(\frac{\Delta N}{\Delta LOGD}\right)_3 = \frac{17.9\ \text{mg/DSCM}}{(5.23599 \times 10^{-10}) \times (1.35\ \text{gm/cc}) \times (7.96\ \text{microns})^3}$$

$$= 5.02 \times 10^7\ \text{particles/DSCM}.$$

For the filter stage

$$\left(\frac{\Delta N}{\Delta LOGD}\right)_F = \frac{8.47\ \text{mg/DSCM}}{(5.23599 \times 10^{-10}) \times (1.35\ \text{gm/cc}) \times (0.231\ \text{microns})^3}$$

$$= 9.72 \times 10^{11}\ \text{particles/DSCM}$$

The test data are usually classified according to sampling location (outlet or inlet), sampling time (day, week, etc.) and combustion chamber or pollution control device conditions (high or low sulfur coal for coal plants, normal or below normal fuel consumption, normal or below normal current density for electrostatic precipitators, etc.). When classified, all of the data taken in a single classification are usually averaged and plotted on appropriate graph paper.

Curves are drawn through the discrete points. At selected particle sizes values of $\Delta M/\Delta \log d$ and $\Delta N/\Delta \log d$ are chosen from these curves for averaging.

Figure C5 shows a typical average $\Delta M/\Delta \log d$ outlet particle size distribution curve, from Andersen impactor data, and Figure C6 shows the same data presented as $\Delta N/\Delta \log d$.

Figure C7 shows similar data for a hypothetical inlet test when a Brink impactor was used. The size distributions of Figures C5 or C7 were used to calculate the penetration/efficiency curve of Figure C8. Confidence limits were calculated for the average data as described in Section C.8.

A computer program has been written which does all of the calculations necessary to generate plotted graphs of the particle size distributions and fractional efficiency curves from the raw field data. Table C4 shows a flow diagram for this program. Copies of the report describing this computer program will soon be available from the EPA or NTIS.

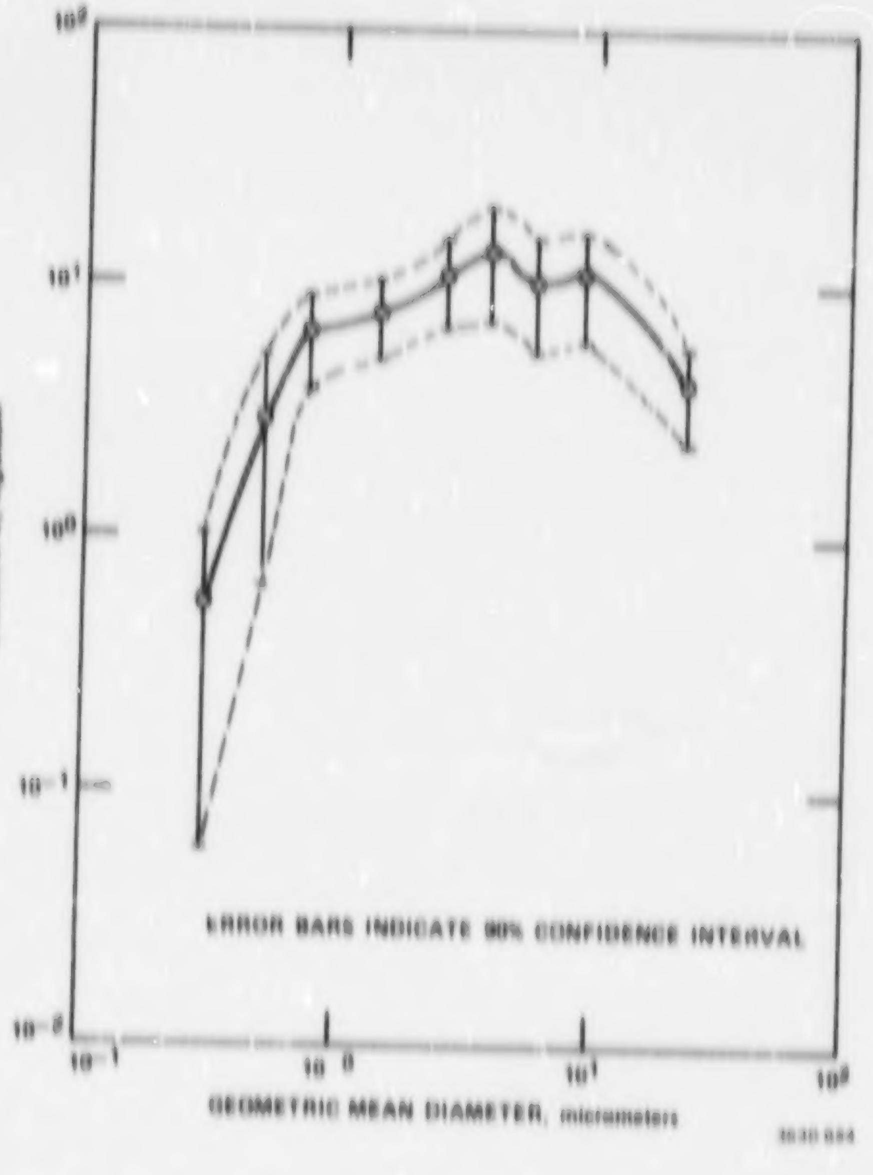

*Figure C5. Hypothetical particle size distribution at an ESP outlet determined from Andersen impactor data.*

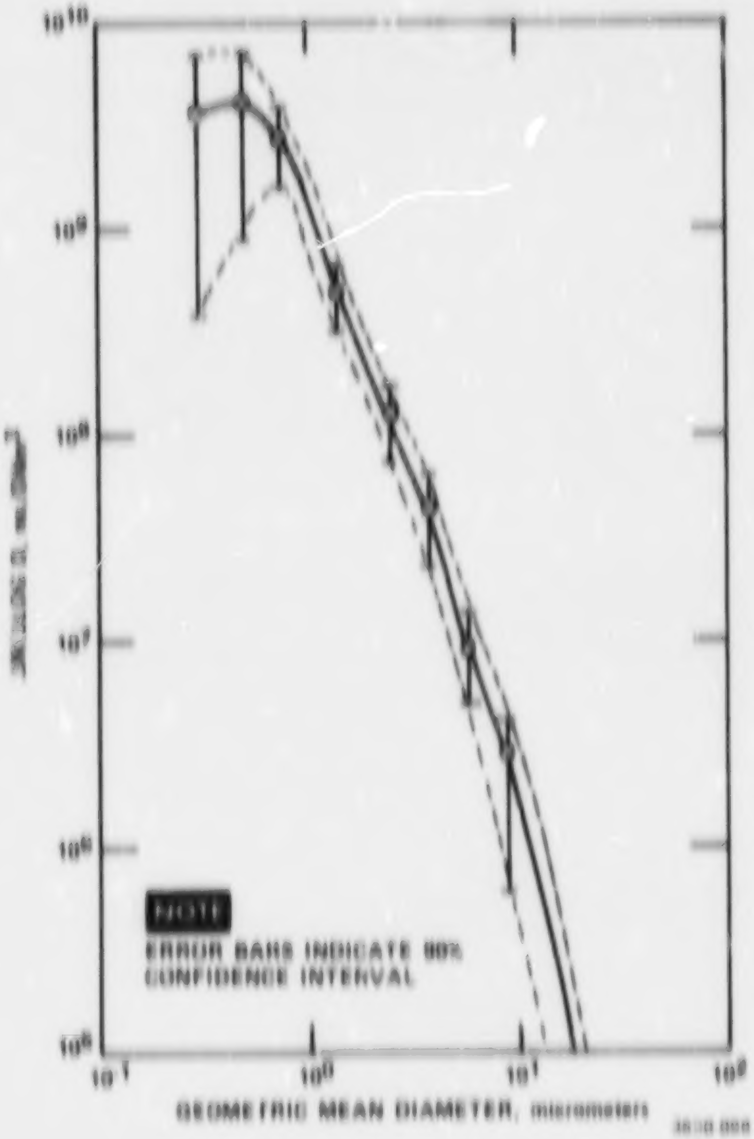

*Figure C6. Hypothetical particle size distribution at an ESP outlet determined from Andersen impactor data.*

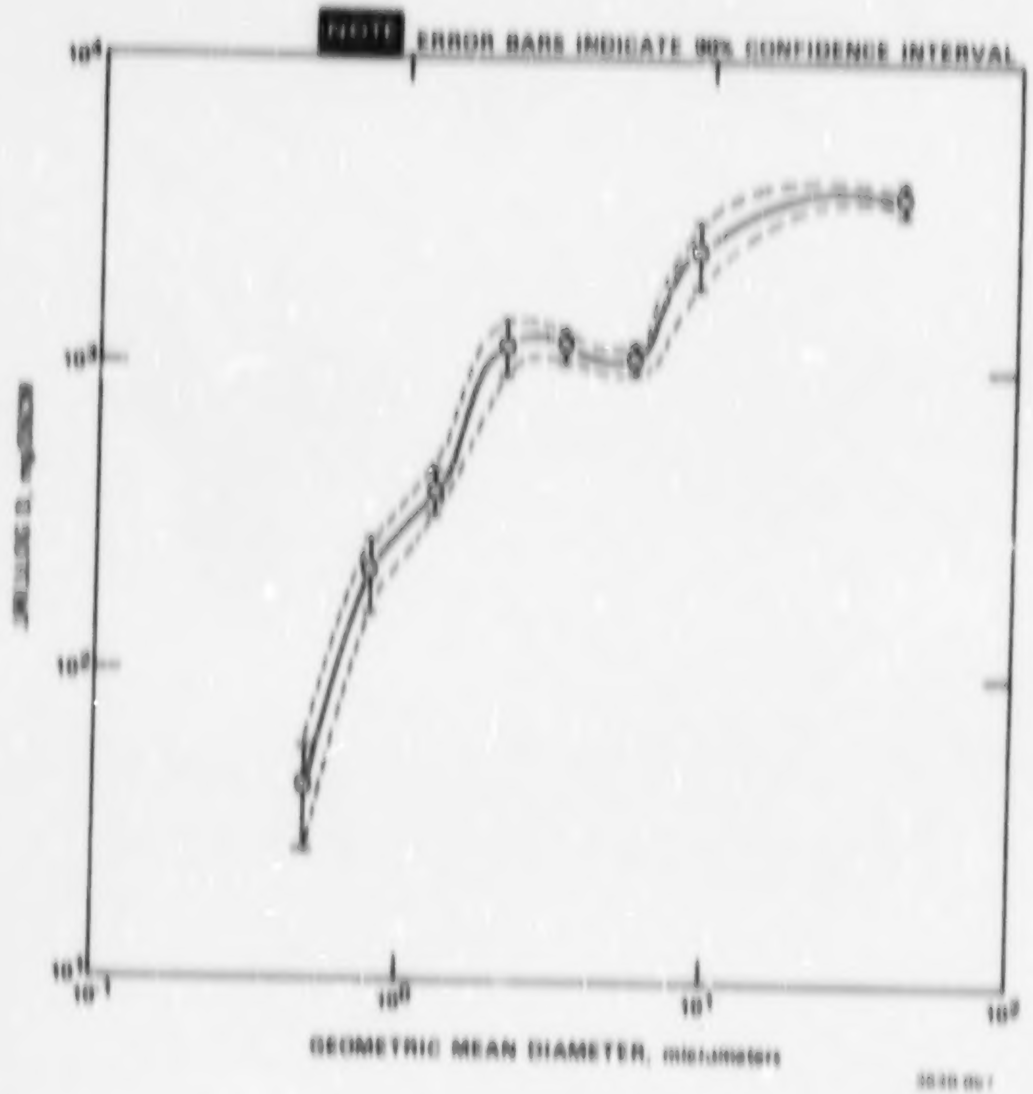

*Figure C7. Hypothetical particle size distribution at an ESP inlet determined from Brink impactor data.*

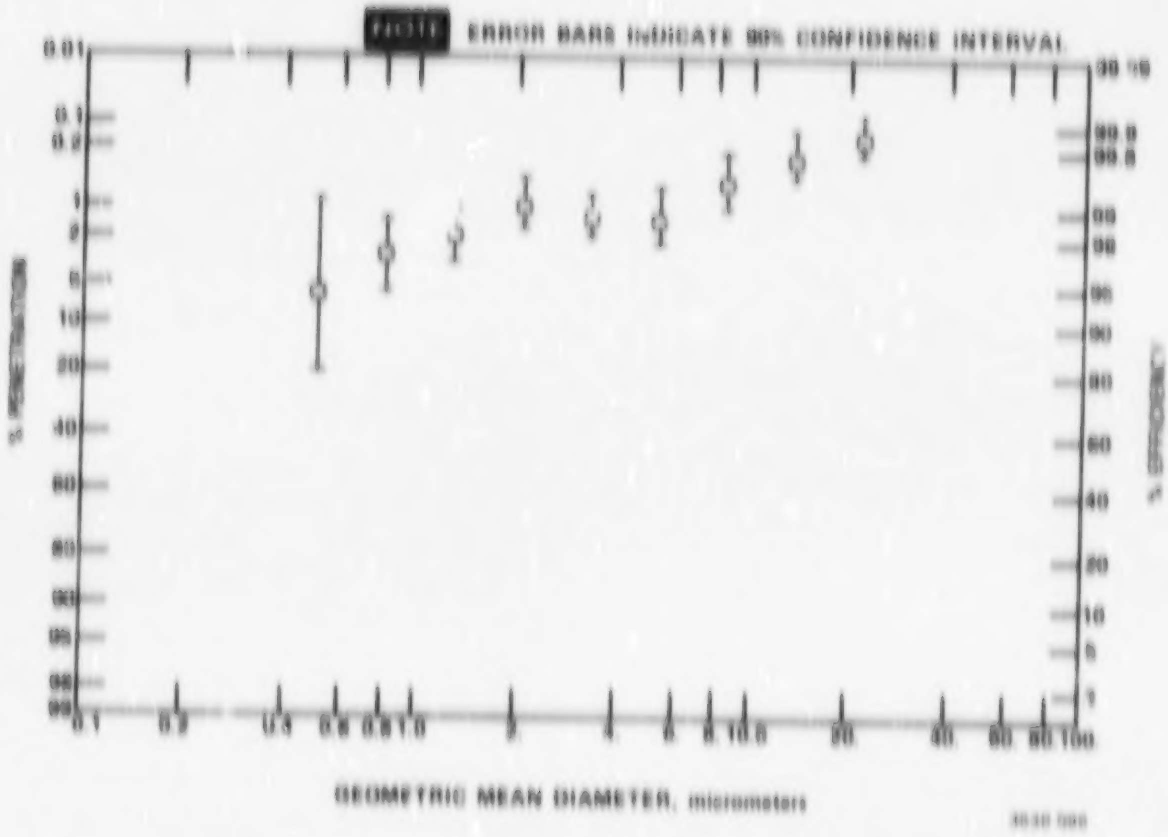

*Figure C8. Hypothetical ESP fractional efficiency curve based on the data presented in Figures C6 and C7.*

# TABLE C4

# PROGRAM FLOW

For all inlet data:

## I. Impactor Program (MPPROG)

Takes testing conditions, stage weights, and impactor constants to produce stage $D_{50}$'s, cumulative and cumulative % mass concentrations < $D_{50}$, geometric mean diameters, and mass and number size distributions.

## II. Fitting Program (SPLIN1)

Uses modified spline technique to fit cumulative mass loading points for each plot. Stores fitting coefficients and boundary points on file.

## III. Graphing Program (GRAPH)

Produces individual run graphs with points based on stage weights and impactor $D_{50}$'s. Also superimposes plot based on fitted data, if desired. Graphs include cum. mass loading, cum. % mass loading, and mass and number size distributions.

## IV. Statistical Program (STATIS)

Recalls cum. mass loading fitting coefficients to produce avg. cum. mass loading, avg. % cum. mass loading, avg. mass size distribution, and avg. number size distribution plots each with 90% confidence bars.

Repeat programs I-IV for outlet data.

## V. Efficiency Program (PENTRA)

Recalls avg. mass size distribution values along with 90% confidence limits for inlet and outlet to plot percent penetration and efficiency with 90% confidence bars.

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# Appendix D

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# APPENDIX D

## SIZE DISTRIBUTIONS OF SUBMICRON AEROSOL PARTICLES

If it is desirable to measure the precipitator collection efficiency for ultrafine particles, measurements of the particle size distribution must be made at the inlet and outlet. This appendix describes procedures for making measurements of the particle size and concentration from 0.01 to 2 μm diameter.

## D.1 SYSTEM FOR EXTRACTIVE SAMPLING

When possible, in-stack sampling is preferred because it eliminates many condensation and sample loss problems which occur when probes are used for extractive sampling. Unfortunately, existing submicron sizing techniques and instrumentation are designed for a laboratory environment and cannot be used in-stack.

Particulate concentrations are usually extremely high in industrial flues and vary by orders of magnitude from one industrial process to another and from the inlet side of a control device to the outlet of the same device. Temperature, pressure, moisture content, and the physical properties of the particulate also vary widely from one industrial process to another. Because of this complexity and the limited useful concentration range for particle sizing techniques, extensive sample dilution and conditioning is required to obtain information on submicron particles in an industrial gas stream.

### D.1.1 Line Losses

When extracting the sample, attention should be given to line losses which can be a problem for particles smaller than 0.01 μm or greater than 1.5-2.0 μm diameter. Since the objective is to measure the concentration of ultrafine particles, there is little interest in measuring particles larger than about 2.0 µm. Thus losses due to impaction and settling are not significant, and isokinetic sampling is unnecessary. Diffusional and electrostatic line losses are of concern, however. For example, at a sampling rate of 1 lpm, a sample line will remove 0.305 µm particles by diffusion at a rate of about 8% of the instantaneous concentration for every foot of sample line, independent of the radius of the sample line. This problem can never be eliminated for the case of out-of-stack sampling, but it can be minimized by (1) using short probes and as high a flow rate as is practical, or (2) by using high, nonturbulent, flow rates through a probe and connecting lines to a conditioning-dilution system in which a sample is split off at the required rate. This second technique allows one to use long probes and change sampling points without disconnecting and reconnecting all sampling lines. Diffusional line losses for non-turbulent flow can be estimated from the equations for diffusion to the walls in a circular geometry (Figure D1). A similar discussion of diffusional line losses has been given by Ensor and Jackson.[1]

### D.1.2 Condensation of Gases

Another problem of concern is condensation. Elements which are at a gaseous state at stack temperature ($SO_3/H_2SO_4$ in particular) can drop below their dew point and form high concentrations of very small particles resulting in anomalously high readings. In the case of $SO_3$ in the presence of $H_2O$, a sulfuric acid fume can be formed if temperatures fall below the acid dew point (temperature, pressure, and concentration sensitive). Once this fume has been formed, very high temperatures are required to re-evaporate the droplets. For this reason temperatures above the dew point must be maintained throughout the system until the gaseous $SO_3/H_2SO_4$ can be removed or diluted. Two techniques appear to be useful for doing this: (1) diffusion to an absorber reagent and (2) dilution of the $SO_3/H_2SO_4$ while hot, to levels at which the mist will not be formed.[1] The main problem with the second technique, however, is that

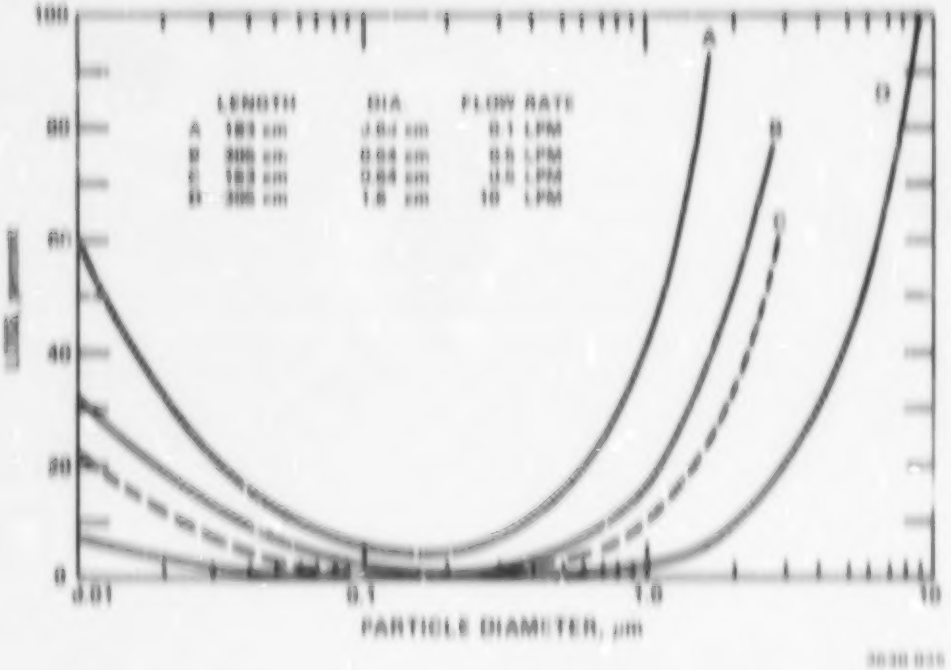

Figure D1. Probe losses due to settling and diffusion for spherical particles having a density of 2.5 gm/cm$^3$ under conditions of laminar flow.

a further dilution is required to bring the aerosol to a temperature within the operating range of the instrument. As a result, the minimum total dilution, which is the product of hot stage and cold stage dilutions, may be excessive for low particulate concentration levels such as those found at the outlet of some gas cleaning devices. Hence the concentration of ultrafine particles even at the minimum dilution may be below the minimum detection level of the sizing instrument.

At low levels of $SO_3$, such as those at power plants burning low sulfur coal, copper has been found to be a successful $SO_3/H_2SO_4$ absorber reagent through the formation of $CuSO_4$. This reagent has the particular advantage that the reaction product is water soluble, and the absorber can easily be rejuvenated. Activated charcoal is also a very effective absorber of $SO_x$, even at relative high concentrations.

A special effort must be made to detect any particles generating interferences and to eliminate these if possible by conditioning the extracted sample. Condensation may be observed by periodically checking the linearity of the dilution system. When the dilution system is adjusted to produce a many-fold change in dilution, the indicated concentration should reflect an equal change in measured concentration.

Figure D2 shows a diffusional absorber/dryer for the removal of water vapor from the sample stream. Figure D3 shows a sample extraction system which includes diffusional absorbers for high temperature use.

### D.1.3 Temperatures

Consideration must also be given to reducing the gas temperature to a level at which the instruments were designed to operate. This is normally done by using a large volume of cool,

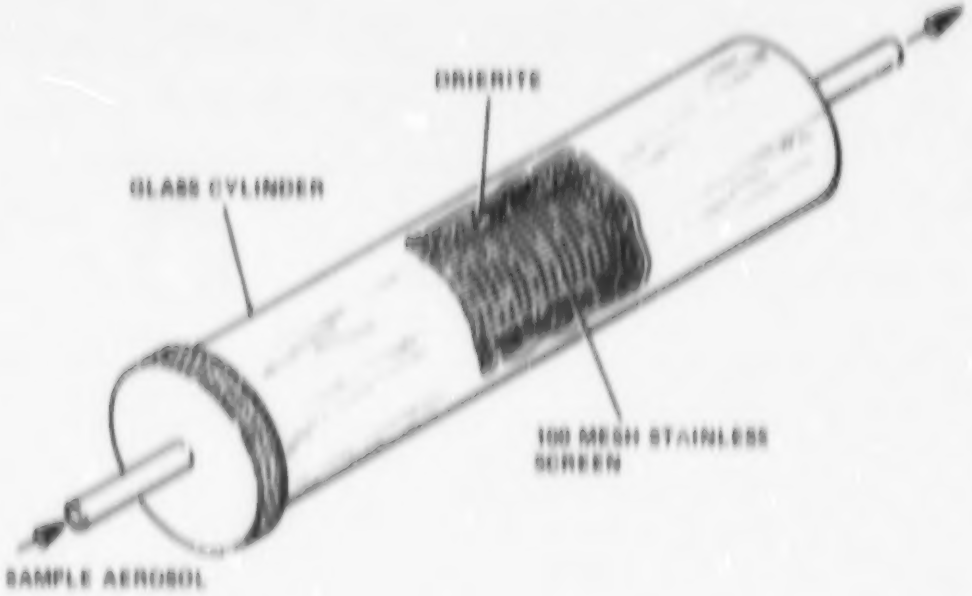
DRIERITE
GLASS CYLINDER
100 MESH STAINLESS SCREEN
SAMPLE AEROSOL
3638 05

*Figure D2. Diffusional absorption apparatus for removal of $H_2O$ from sample aerosol.*

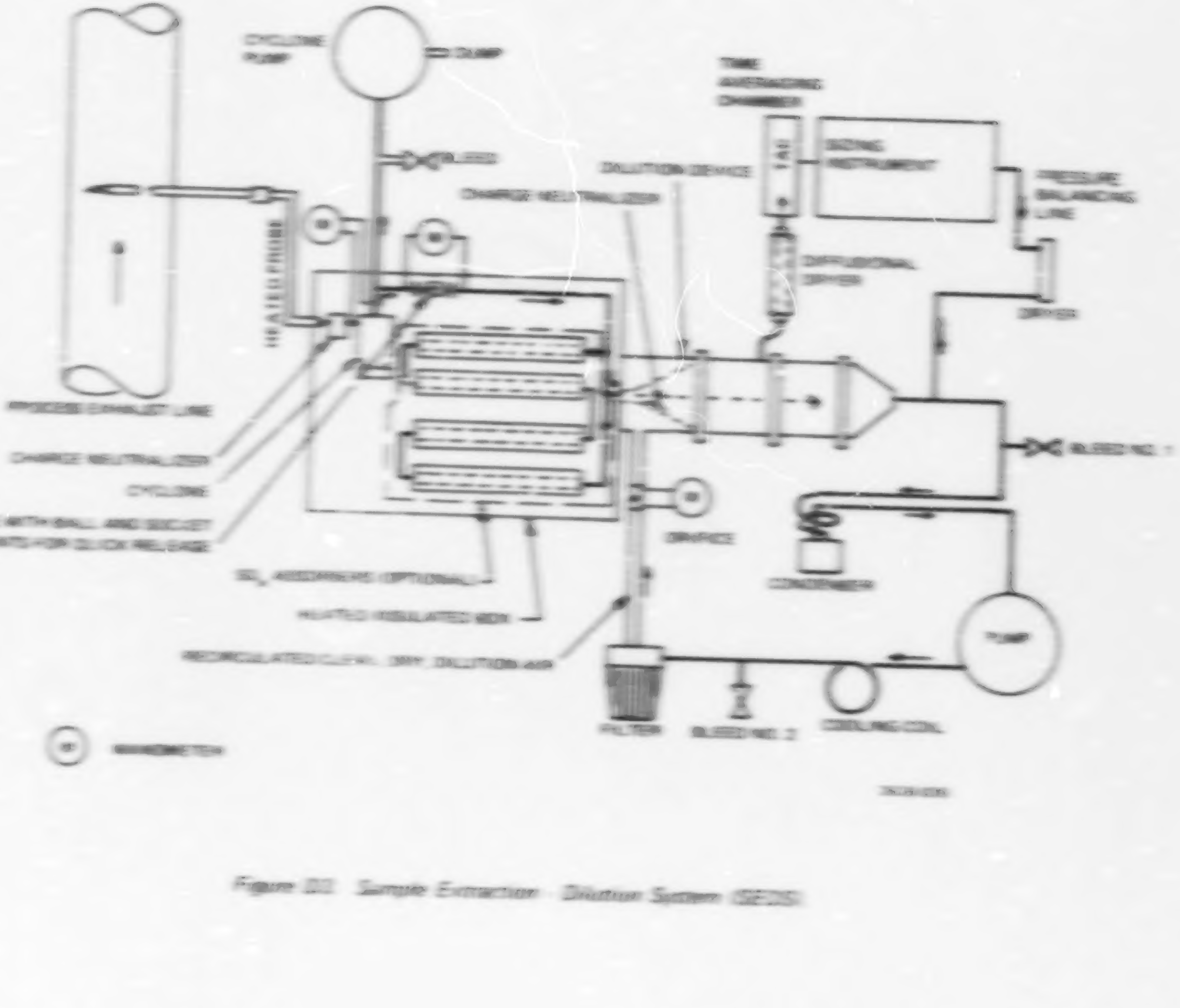
PROCESS EXHAUST LINE
CYCLONE PUMP
DUMP
BLEED
HEATED PROBE
M
CHARGE NEUTRALIZER
CYCLONE
ORIFICE WITH BALL AND SOCKET JOINTS FOR QUICK RELEASE
$SO_x$ ABSORBERS (OPTIONAL)
HEATED INSULATED BOX
RECIRCULATED CLEAN, DRY, DILUTION AIR
FILTER
BLEED NO. 2
COOLING COIL
PUMP
CONDENSER
ORIFICE
CHARGE NEUTRALIZER
DILUTION DEVICE
TIME AVERAGING CHAMBER
TAC
SIZING INSTRUMENT
PRESSURE BALANCING LINE
DIFFUSIONAL DRYER
DRYER
BLEED NO. 1
MANOMETER

Figure [illegible]. Sample Extraction - Dilution System (SEDS) dry dilution air but in some cases can be done by simply pulling the sample through an ice bath condenser. It should be remembered, however, that unless condensible gases have previously been removed, it is possible to form a condensation fume when using a simple condenser.

### D.1.4 Electrostatic Losses

Electrostatic line losses can also be a problem. Due to charges present on the particles, large static electric fields can be established which result in particle deposition and nonrepresentative sampling. Line losses may be more severe if the aerosol particles are charged. Electric fields may exist which result in particle deposition and nonrepresentative sampling. This problem is most severe if the sample lines are made of insulating materials. Particle losses may also be a problem in the diffusion batteries where the theory assumes that the particles are electrically neutral and no consideration is given to unknown electrical forces. Also, it is assumed in the application of the electrical aerosol analyzer that the sample aerosol particles initially bear no charge. Precharged particles could acquire charges different from those of calibration, causing them to exhibit different mobility vs size characteristics. It is desirable to neutralize the particle charge prior to entering the probe nozzle, and charge neutralization to Boltzmann Equilibrium can be accomplished by exposure to an ion field created by radioactive materials, however, no suitable radioactive source has been developed for in-stack applications. The approach generally taken has been to use radioactive materials such as $P_o^{210}$ in the diluter to neutralize the particulate after it has been cooled and before it goes to the sizing instruments. The extent of electrostatic interferences, however, has not been well quantified.

### D.1.5 Humidity

High humidities can alter the charging characteristics of electrical mobility analyzers and can cause water to condense on particles and change their size (similar to the controlled process occurring in a condensation nuclei counter). Humidity problems can be eliminated by the use of (1) dry dilution air or (2) diffusional dryers.

### D.1.6 Dilution

In-stack concentrations generally exceed instrument concentration limits and must be reduced to levels at which the instrument functions properly. Large changes in the aerosol size distribution due to coagulation are also of concern, particularly in a diffusional configuration involving long residence times, since the loss rate due to coagulation for a given size particle rises rapidly with increasing concentration.

Most of the dilution systems used to date involve the measurement of two different flow rates. Several methods are available for doing this: (1) Rotameters, (2) Orifices, (3) Venturis, and (4) Mass Flowmeters. Spink[1] has summarized the use of the first three and Parry[2] has described the use of several mass flowmeters in an automated system. Rotameters are convenient for measuring cool dilution air flow rates but could cause sample losses when measuring sample gas flows. They are also sensitive to temperature, pressure, and gas composition. Flowmetering orifices are useful for measuring flow rates of hot gases but significant sample losses can occur for particles larger than about 2 um. Orifices also require, as do venturis, that pressure drops, etc. be monitored in order to calculate the flow rate. Venturis have the advantage that less turbulence occurs in the meter and hence size dependent losses should be much less than those for orifices. Venturis have the disadvantage that the pressure tap must be taken at the Vena Contracta making them difficult to construct for low flow rates. Mass flowmeters are ideal but may require line restrictions in order to throttle the flow rate.

### D.1.7 Sample Extraction-Dilution Systems (SEDS)

Figure D3 is a block diagram of a sample extraction-dilution system (SEDS) developed by Southern Research Institute under EPA Contract No. 68-02-3114. In this system a 233 cm³/sec sample flow is removed from the process exhaust stream and is pulled through a rigid probe, a flexible connector hose, and a cyclone, into a "T" where the flow splits. The excess flow is dumped and the desired sample flow goes into the diluter via a calibrated orifice and an optional bank of sulfur oxide absorbers. The cyclone, orifice, and sulfur oxide absorber bank are housed in a heated box so that all components of the system except the diluter can be maintained at the stack temperature (up to 200°C) to prevent condensation. Pressure taps for the cyclone and the orifice allow continuous monitoring of the cyclone flow rate and the orifice flow rate by reading the pressure drop across the respective component.

Currently, charge neutralization is done in the cone of the diluter by two 500 µC Polonium-210 strips, mounted as shown. The sample gas enters the dilution chamber at the apex of a perforated cone into which clean, dry air is pumped through the perforations, creating a highly turbulent mixing zone. Calibration data for the diluter, Figure D4, is shown in Figures D5 and D6. At a downstream point, after adequate mixing has occurred, the diluted sample is extracted and conveyed to the sizing instrument. This diluted sample passes through a diffusional dryer where any remaining moisture is removed. The major drying action is accomplished by using dilution air which has been passed through an ice bath condenser.

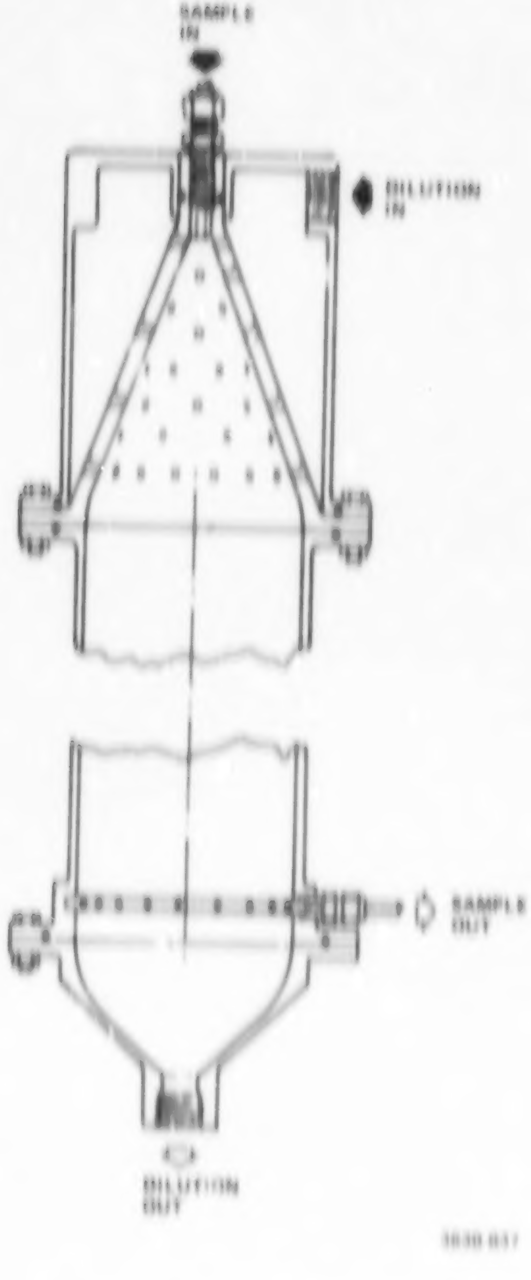
SAMPLE IN
DILUTION IN
SAMPLE OUT
DILUTION OUT
3630.837

*Figure D4. Sample Extraction Diluter, cut-away view.*

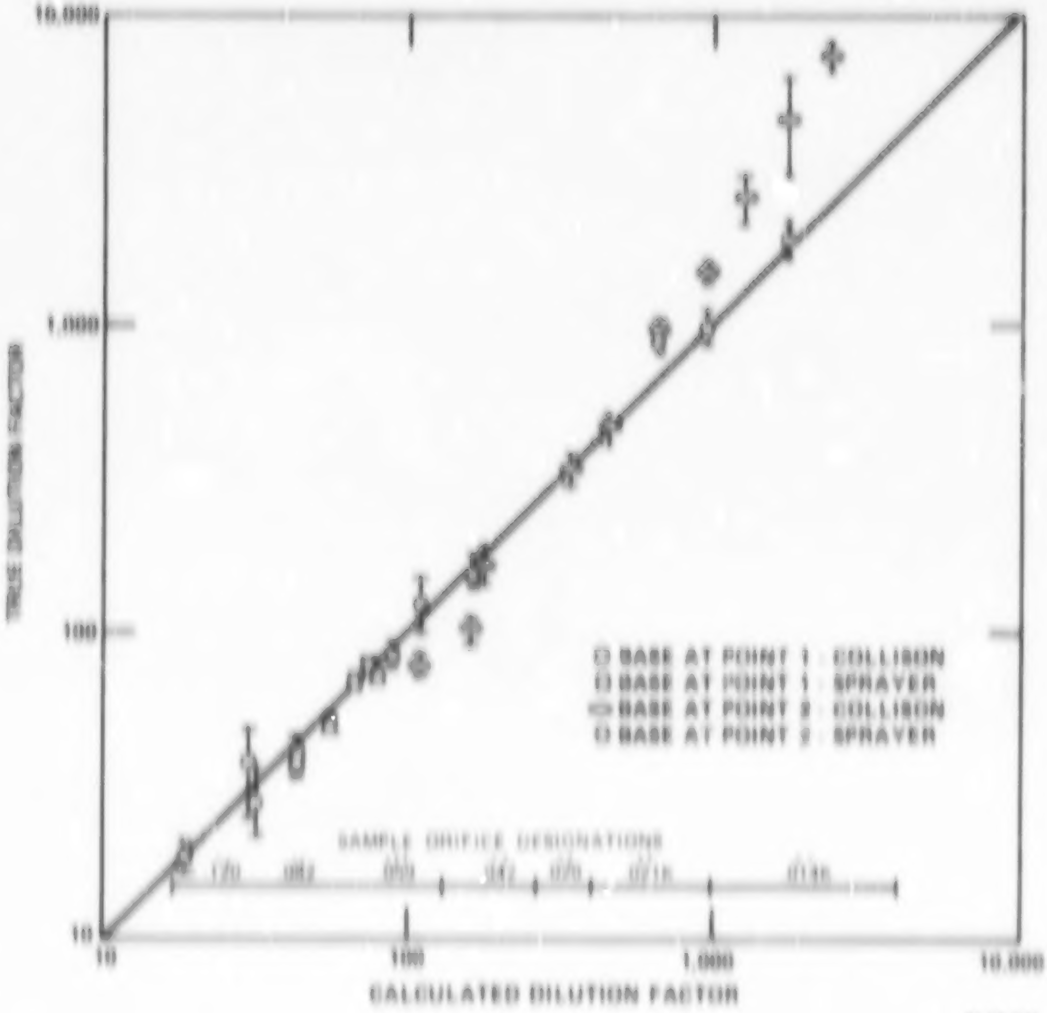

*Figure D5. Calculated dilution versus true dilution for the Southern Research Institute Ultrafine Particle Diluter, 0.092 μm particles.*

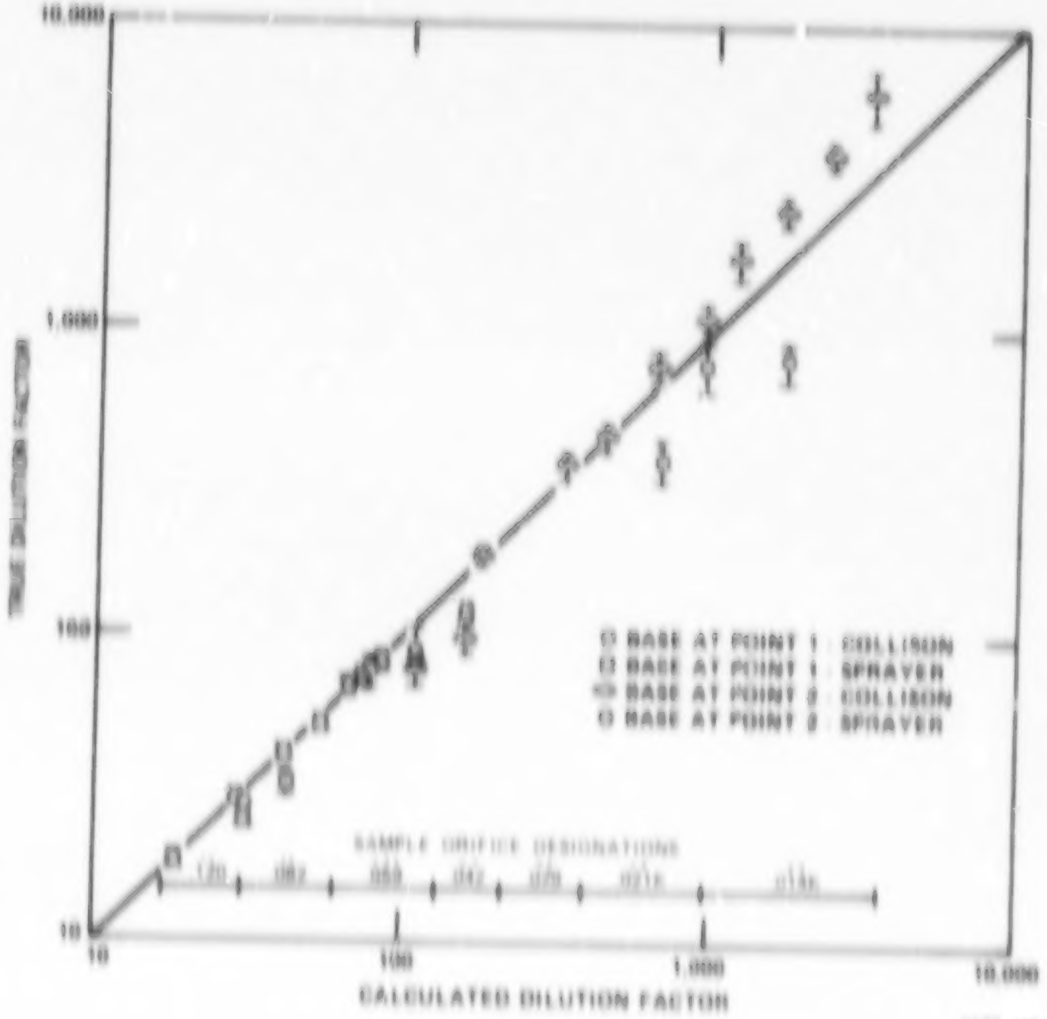

*Figure D6. Calculated dilution versus true dilution for the Southern Research Institute Ultrafine Particle Diluter, 0.15 μm particles.*

Just prior to entering the sizing instrument, the sample passes through a plenum to damp out short term concentration changes. The exhaust from each instrument is returned to the diluter to reduce pressure drops across the sizing devices. If further drying of this recycled gas is necessary, absorption driers are placed in the instrument exhaust lines.

Changing the sample air flow and the dilution air flow allows one to change the dilution ratio. Sample air and dilution air flowrates are controlled by two bleed valves on the dilution air pump, one upstream of the pump (#1) and one downstream (#2). Manipulation of these valves changes the internal pressure of the diluter which, in turn, sets the sampling rate. As the pressure in the diluter is reduced, the sample flow rate is increased. In practice, the operation of these valves changes the dilution air flow only about 10% for a many-fold change in sample flow.

McCain[1] has reported some problems with an earlier prototype conditioning system; growth of particles when high concentrations of $SO_3$ are present, pluggage of orifices, single point sampling limitations, and diffusional losses in sampling lines. The configuration described above allows the use of optional $SO_x$ absorber chambers, rapid replacement for plugged orifices, quick positive determination of partial pluggage, full traverse sampling capability, and decreased sample line losses. The hot box configuration also increases the total time available for data acquisition by decreasing the time needed to change orifices or dilution ratios (2 minutes compared to about 30 minutes).

Different sampling and dilution systems have been developed and reported by Ensor and Jackson,[1] Bradway and Cass,[4] and Schmidt et al.[7]

It is clear that the most difficult problem in making size distribution measurements of submicron particles is extracting a representative sample from the duct and conditioning it for compatibility with the sizing instruments.

A variety of instruments are available to characterize the conditioned sample, and these are described in the remainder of this appendix.

## D.2 SUBMICRON PARTICLE SIZING TECHNIQUES BASED ON PARTICLE DIFFUSIVITY

### D.2.1 Diffusion Batteries

Fuchs[9] has reviewed diffusional sizing work up until 1956, while Sinclair,[10,11] Breslin *et al.*,[12] Twomey,[13] and Sansone and Weyel[14] have reported more recent work, both theoretical and experimental.

Diffusion batteries may consist of a number of long, narrow, parallel channels, a cluster of small bore tubes, or a series of screens. Variations in the length and number of channels (tubes, or screens) and in the aerosol flow rate are used as means of measuring the number of particles in a selected size range. As the aerosol moves in streamline flow through the channels, the particles diffuse to the walls at a predictable rate depending on the particle size and the diffusion battery geometry. It is assumed that every particle which reaches the battery wall will adhere; therefore, only a fraction of the influent particles will appear as the effluent of a battery. It is only necessary to measure the total number concentration of particles at the inlet and outlet to the diffusion battery under a number of conditions in order to calculate the particle size distribution.

When the Stokes diameter is used to describe particle size, the penetration of diffusion batteries is virtually independent of physical properties of the individual aerosol particles.

*Parallel plate geometry* - The parallel plate geometry is convenient because of ease of fabrication and the availability of suitable materials, and also because sedimentation can be ignored if the slots are vertical, while additional information can be gained through settling if the slots are horizontal. See Figure D7. Disadvantages of the parallel plate diffusion batteries are (1) the bulk of the diffusional batteries, and (2) the long transport time required to measure a size distribution.

The mathematical expression for the penetration of a rectangular slot or parallel plate diffusion battery by a monodisperse aerosol was given in series form by Gormley and Kennedy.[11] The coefficients were calculated and tabulated by Twomey[11] using a computer.

By varying the number of diffusion batteries in series and the flow rate, it is possible to measure penetrations under a variety of conditions. Using a set of diffusion batteries such as those developed at Southern Research (four-98 channel diffusion batteries, and a 13 channel diffusion battery) and measuring the penetration at three different flow rates, yields fifteen data points from which the particle size distribution (0.01-0.2 μm diameter) can be reconstructed.

When calculating the fraction ($n/n_0$) of the aerosol which penetrates a series of diffusion batteries, the transport time through the diffusion batteries must be taken into account. This transport time is about 1½-5 minutes for a 98 channel,

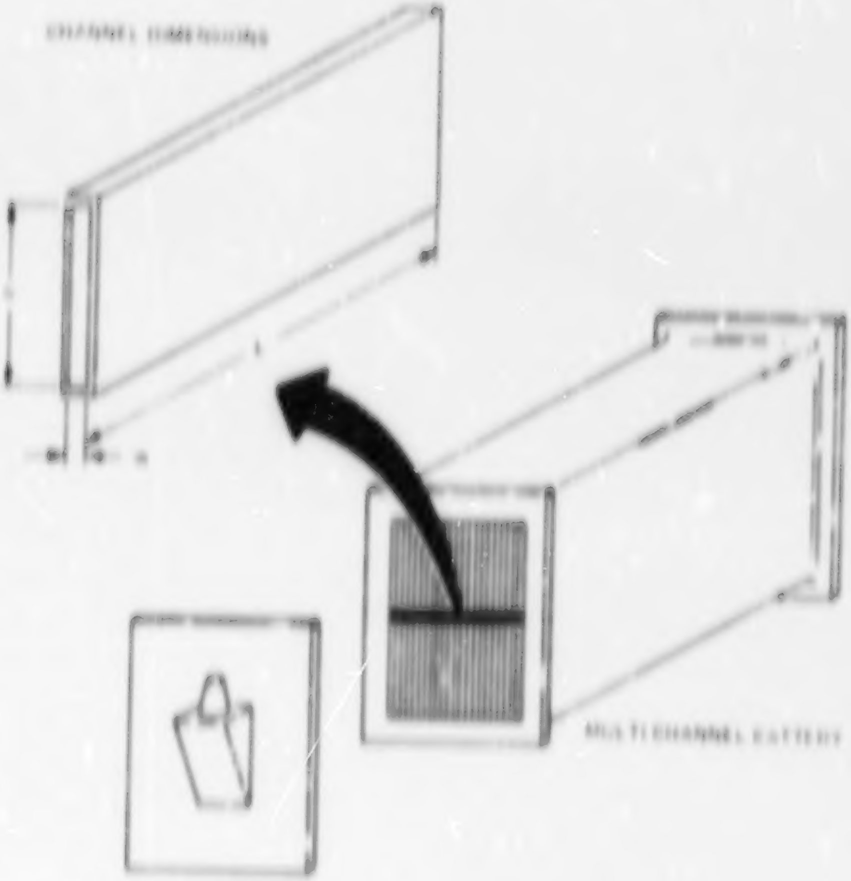
CHANNEL DIMENSIONS
MULTICHANNEL BATTERY

Figure D7. Parallel Plate Diffusion Battery.

parallel plate diffusion battery. Thus, with four of these diffusion batteries in series, n, at a particular time, would be related to $n_0$ at a point 16-20 minutes earlier in time on a chart recording. Making a complete measurement of a particle size distribution requires 2-4 hours and diffusional measurements are most useful on stable sources where the distribution is constant in time when using parallel plate diffusion batteries. The transport time is determined by the open air volume of the diffusion battery and flow rate. If the same geometry constant can be obtained with smaller open air volumes, the transport time can be reduced. This can be accomplished by decreasing the plate spacing, but one very quickly approaches problems with material flatness errors, or equipment survivability when such materials as precision ground graphite sheets are used. These problems seem to have been eliminated with the screen geometry, which is discussed below.

Screen geometry - Sinclair[11] and Breslin et al[11] report success with more compact, tube-type and screen-type arrangements in laboratory studies.

Although the screen-type diffusion battery must be calibrated empirically, it offers convenience in cleaning and operation, and compact size. Figure D8 shows Sinclair's geometry. This battery is 21 cm long, approximately 4 cm in diameter, and weighs 0.9 kg.

This system is commercially available from Thermosystems, Inc., St. Paul, MN 55113 as the Model 3040 Diffusion Battery. Because of the small internal volume of the battery, the time necessary to obtain a test on one battery is reduced by about a factor of ten as compared to the parallel plate batteries. This diffusion battery system allows data to be collected on process streams where the particle concentration is somewhat unstable (~15 minutes per cycle).

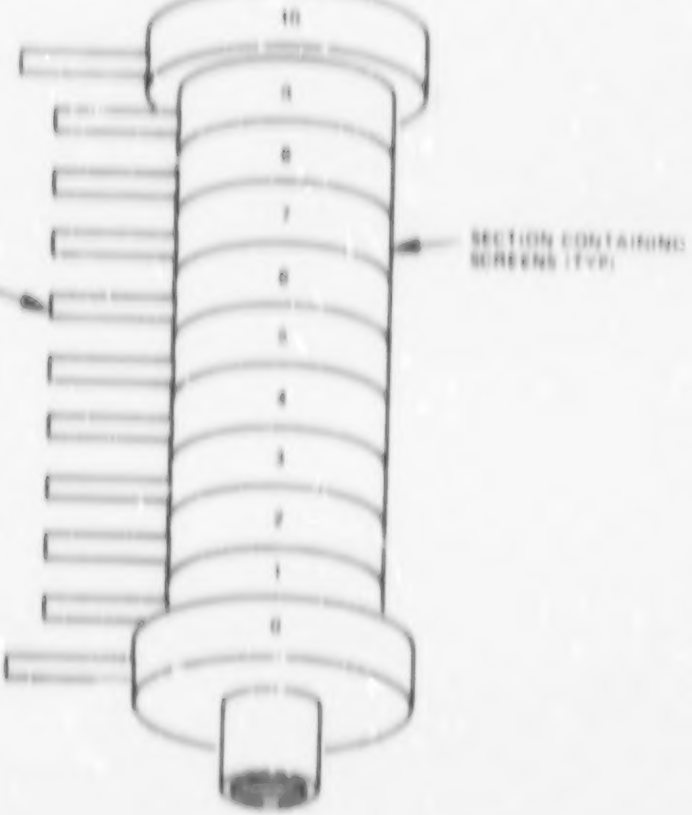
10
9
8
7
6
5
4
3
2
1
0
SAMPLING PORT (TYP)
SECTION CONTAINING SCREENS (TYP)

*Figure D8.* *Screen type diffusion battery. The battery is 21 cm long, 4 cm in diameter, and contains 55 635 mesh stainless steel screens.*

A disadvantage of the small volume is the sensitivity to surges in flow rate caused by the commercially available CN counter.

### D.2.2 Particle Concentration Indicators-Condensation Nuclei Counters (CNC)

Condensation nuclei counters function on the principle that particles act as nuclei for the condensation of water or other condensable vapors in a supersaturated environment. This process is used to detect and count particles in the 0.002 to 0.3 micron range (often referred to as condensation or Aitken nuclei). In condensation nuclei detectors, a sample is withdrawn from the gas stream, humidified, and brought to a supersaturated condition by reducing the pressure. In this supersaturated condition, condensation will be initiated on all particles larger than a certain critical size and will continue as long as the sample is supersaturated. This condensation process forms a homogeneous aerosol, predominantly composed of the condensed vapor containing one drop for each original particle whose size was greater than the critical size appropriate to the degree of supersaturation obtained; a greater degree of supersaturation is used to initiate growth on smaller particles. The number of particles that are formed is estimated from the light scattering properties of the final aerosol.

Because of the nature of this process, measurements of very high concentrations can be in error as a result of a lack of correspondence between particle concentration and scattering or attenuation of light. Additional errors can result from depletion of the vapor available for condensation. Certain condensation nuclei measuring techniques can also obtain information on the size distribution of the nuclei; that is, variations in the degree of supersaturation will provide size discrimination by changing the critical size for which condensation will occur. However, MacLauren and Junge[15] have predicted that the critical size for initiating condensation is also affected by the volume fraction of water soluble material contained in the original aerosol particle, so the critical size will be uncertain unless the solubility of the aerosol particles is known. At very high degrees of supersaturation (about 400%), solubility effects are only minor and essentially all particles in the original aerosol with diameters larger than 0.002 μm will initiate the condensation process.

A continuous flow CN counter has been described by Sinclair[16] and an absolute calibration of a CN counter has been done by Liu.[17] The theory and principles of operation of CN counters has been described by Haberl.[18,19]

### D.2.1 Using Diffusion Batteries With Condensation Nuclei Counters and a Sample Extraction-Dilution System To Measure Concentions of Submicron Aerosols In Industrial Flue Gases

Before taking equipment into the field, a preliminary examination of the sampling site should be made. There must be ample space for the diffusion batteries (D.B.) and the condensation nuclei counter (CNC). The sample lines to and from the D.B.'s should be as short as is practical. The diffusion batteries should be placed out of direct sunlight to reduce thermal interferences.

Four models of CNC's are presently used. Two automatic models are the General Electric (GE)* and the Environment One

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*General Electric - Ordnance Systems, Electronics Systems Division, Pittsfield, MA 01201.

Model Rich 100 (E-1).** Small manual particle detectors are also commercially available from Gardner Associates*** and Environment One.** The valving system in the GE is mechanical, and pressure differentials across the valves are irrelevant. However, the E-1 has pneumatic valves and a pressure difference of greater than 2 inches of water across the valves will lock them either open or shut, thus the return lines on the E-1 CNC must be connected to the sample extraction system to prevent instrument malfunctions. A return line is normally used on the GE but may not be connected to the diluter in some circumstances. The GE creates substantial pulsation in the sample lines which is intolerable for diffusional analysis, and an antipulsation device consisting of two metal cylinders connected by a small orifice may be used as a pneumatic R-C network to damp the oscillations to an acceptable level.

Once all sample and return lines have been connected, the equipment is turned on and allowed to warm up. The flows are then adjusted to the proper rates. If the GE model is used, the vacuum gage on the front panel should read 8 inches of Hg as recommended in the operation manual. This gives a nominal flow rate of 6 lpm and a sample supersaturation after expansion of approximately 400%. The E-1 may be adjusted to any desired flow rate between about 0.6 lpm and 4.2 lpm.

The water supplies for the humidifiers are filled with a mixture of distilled water and a wetting agent. About 0.5% Kodak photoflow (ethylene glycol) is used in the water. This reduces the surface tension of the water and allows the wick to wet better and more quickly.

---

**Environment-One Corporation, Schenectady, NY 12301.

***Gardner Associates, Schenectady, NY 12301.

Before any data can be taken the system should be leak checked. This is done by connecting all the D.B.'s in series, clamping one end off and pumping several inches of mercury vacuum. If the vacuum holds data can be taken. If there is a leak it can be isolated by repeating the process with successively fewer D.B.'s until the leaky one is found. The exact location of the leak can be determined by putting a concentrated condensation nuclei source (e.g., a burning string) close to various parts of the D.B. When the leak is found, the CNC panel meter will rapidly rise.

There are two methods of data collection: graphing the CNC output on a strip chart recorder or directly writing down the meter reading. In general, a combination of the two is used to insure that no faulty connection exists between the CNC and the chart recorder or that the chart recorder is malfunctioning.

Since with either the GE or the E-1 CNC the largest flow possible is 6 lpm, a certain minimum amount of time is required to pull the sample through the parallel plate D.B.'s. If graphical techniques are used a characteristic output will be observed. The indicated concentration is zero while the clean air already in the D.B.'s is being exhausted. Then the output rises to a peak and stabilizes. It is at this point that meaningful data is being taken. However, if data is taken by meter readings alone this characteristic response is very difficult to follow. Adequate time must be allowed for transport through the D.B.'s before each reading is taken. These transport times have been calculated for 6 lpm for two types of parallel plate D.B.'s used: Thirty seconds for the 13 channel parallel plate and five minutes for each 98 channel parallel plate D.B. used. Quite frequently the in-stack concentrations are unsteady and data is normally recorded for several minutes after the system has had time to stabilize as a double check on the validity of the data and to provide some time averaging of the data on fluctuating sources. With the screen type diffusion batteries, transport times of about 1/10 of the times given above can be used.

If the GE CNC is used, diffusional sizing cut points are selected by changing the number of D.B.'s, since the flow rate is constant. However, with the E-1, the cut sizes may be adjusted by either changing the number of D.B.'s or the flow rate. The flow rate is sometimes held constant because source fluctuations introduce so much uncertainty that attempts to achieve high resolution are futile.

In order to obtain a set of data, the CNC is first connected directly to the diluter to obtain total concentration. Data is then taken by pulling the sample next through a single battery, two batteries, three batteries, etc., until all the available permutations of geometry and flow rate have been used.

If only one set of equipment is available, after data are collected at the inlet the equipment is carried to the outlet, set up, leak checked, and the above procedure is repeated.

### D.2.4 Data Reduction Techniques and a Sample Calculation

Fuchs et al[11] presented a technique for calculating the particle size distribution from raw data, assuming that the size distribution was log normal. A technique suggested by Sinclair[7] does not include this restriction. In Sinclair's method a nomograph is prepared using the penetration for each diffusion battery geometry and flow rate and a large number of monodisperse particle sizes. Comparing this nomograph with experimental penetrations, one calculates the particle size distribution using a "graphical stripping" process. However, it is usually more convenient to use a "$D_{50}$" technique like that used for the reduction of cascade impactor data.

Data for a given test condition are averaged as follows to yield a representation of the source at that condition. All instrument readings are converted to indicated concentrations by means of individual instrument calibration curves. These values are then corrected for dilution to obtain flue gas concentration. Since the CNC calibration curve is non-linear, multiplying by the dilution factor before converting to concentration will yield erroneous results. Next, process averaging is accomplished by taking appropriately weighted averages of the data obtained through the various process cycles. These weighted averages are found for each D.B. arrangement, including no D.B.'s, and test condition, both inlet and outlet.

For the sample calculation it is assumed that five parallel plate diffusion batteries were used, in four configurations, and at three flow rates to obtain data at twelve $D_{50}$ sizes. These five diffusion batteries consisted of one 17 channel (Type A) and four 98 channel (Type B) units. The four sampling configurations were 1) one Type A, 2) one Type B, 3) two Type B in series, and 4) four Type B in series. These diffusion batteries are similar to the one depicted in Figure D7. The three flow rates were 1, 6, and 10 liters per minute. The aerosol was sampled from a Sample Extraction and Dilution System and the total number of particles entering and exiting the diffusion batteries was determined using a condensation nuclei counter (CNC). The sample data for this experiment is shown in Table D1.

To calculate the particle size for 50% penetration (the $D_{50}$) through a diffusion battery configuration, the following equations must be used.

The penetration of a rectangular plate diffusion battery is given by

$$P = \frac{n}{n_0} = \left[0.9104e^{-1.88522DY} + 0.0531e^{-21.431DY} + 0.015e^{-62.317DY} + 0.0068e^{-124.537DY}\right]^M \qquad (D1)$$

where D = Particle diffusivity, cm²/sec,
Y = Diffusion battery flow rate-geometry constant, sec/cm²,
m = Number of identical batteries in series,
$n_0$ = Diffusion battery inlet concentration, #/cm³, and
n = Diffusion battery outlet concentration, #/cm³.

The particle diffusivit/, D, is given by

$$D = kTB \quad , \quad \text{where} \qquad (D2)$$

k = Boltzmann's Constant, gm cm²/sec² °K,
T = Absolute Temperature, °K, and
B = Particle Mechanical Mobility, sec/gm.

The particle mechanical mobility is given by

$$B = \left[1 + 2.49\,(\lambda/d) + .84\,(\lambda/d)e^{-(.44)(d/\lambda)}\right]/3\pi\mu d \qquad (D3)$$

where λ = gas mean free path, cm,
d = particle diameter, cm, and
μ = gas viscosity, gm/sec cm.

The gas mean free path is given by

$$\lambda = 3.109 \times 10^{-4}\,\frac{\mu}{p}\,\sqrt{T/M} \qquad \text{where} \qquad (D4)$$

# TABLE D1

## DIFFUSION BATTERY SAMPLE CALCULATION DATA

## CNC

| Configuration | (ℓ/min) | Calc. $D_{50}$ (μm) | Reading (#/cm³) | Dilution Factor | Actual Concentration (#/cm³) |
|---|---|---|---|---|---|
| 1 Type A | 6 | 0.02 | 53000 | 1000 | 53.0 x $10^6$ |
| 1 Type B | 6 | 0.06 | 43000 | 500 | 21.5 |
| 2 Type B | 6 | 0.098 | 45000 | 200 | 9.0 |
| 4 Type B | 6 | 0.17 | 11000 | 200 | 2.2 |
| 1 Type A | 1 | 0.028 | 84000 | 500 | 42.0 |
| 1 Type B | 1 | 0.07 | 33600 | 500 | 16.8 |
| 2 Type B | 1 | 0.12 | 29000 | 200 | 5.8 |
| 4 Type B | 1 | 0.20 | 5500 | 200 | 1.1 |
| 1 Type A | 10 | 0.015 | 64000 | 1000 | 64.0 |
| 1 Type B | 10 | 0.045 | 62000 | 500 | 31.0 |
| 2 Type B | 10 | 0.08 | 66500 | 200 | 13.3 |
| 4 Type B | 10 | 0.14 | 20500 | 200 | 4.1 |

$P$ = Ambient pressure, atm,
$T$ = Absolute temperature, °K, and
$M$ = gas mean molecular weight, gm/gm-mole.

The approximate gas viscosity is given by

$$= \left[(.495\ (T-294^\circ K) + 182)\right] \times 10^{-6} \text{ gm/cm sec.} \qquad (D5)$$

The diffusion battery flow rate-geometry constant, Y, is given by

$$Y = \frac{4LhN}{QW} \text{ where} \qquad (D6)$$

$L$ = Channel length, cm,
$h$ = Channel height, cm,
$N$ = Number of channels,
$Q$ = Flow rate, cm$^3$/sec, and
$W$ = Channel width, cm.

See Figure D-7.

The two diffusion batteries under consideration in this example have the following dimensions.

| | Type A | Type B |
|---|---|---|
| L | 45.72 cm | 45.72 cm |
| h | 10.15 cm | 11.46 cm |
| W | 0.10 cm | 0.10 cm |
| N | 13 | 98 |

Thus, $Y_A = 2.358 \times 10^3$ sec/cm$^2$ and $Y_B = 2.006 \times 10^4$ sec/cm$^2$ for $Q$ = 6 l/min.

The manipulation of these equations allows one to calculate the penetration of the diffusion batteries at different flow rate and particle-size combinations. After plotting the penetration versus particle size, the $D_{ij}$'s can be determined.

To aid in the calculation of the particle diffusivity and penetration of a particular D.B. arrangement, two programs have been written for the Hewlett-Packard HP-65 Programmable Calculator. If a programmable calculator is not available, the calculations can be done manually using the equations given above. The calculator programs and their applications are described below:

Program 1 is used to calculate the viscosity ($\mu$) and mean free path ($\lambda$) of standard air. These values are then used to calculate the diffusivity (D) for a monodisperse aerosol having diameter d. Given values for the flow rate-geometry configuration (y) and the diffusivity (D), Program 2 is used to calculate the theoretical penetration ($n/n_o$).

Program 1: Diffusivity (Program Steps are Listed in Table D2.)

Over the temperature range from 0-350°C, the viscosity of dry standard air is very nearly linear. For a given temperature $\mu$ can be found, in CGS units, from:

$$\mu = \left[.495\ (T - 294°K) + 182\right] \times 10^{-6} \text{ poise}$$

Knowing the viscosity, the mean free path is given by

$\lambda = 5.77 \frac{\mu}{P}\sqrt{T} \times 10^{-4}$ cm for standard air (P is the absolute pressure, atm.).

From a knowledge of μ and λ for the carrier gas, the diffusivity ($D_i$) of a monodisperse aerosol having diameter ($d_i$) is given by the following equation:

$$D_i = (1.46 \times 10^{-17}) \frac{T}{\mu d_i} \left[ 1 + 2.49 \left(\frac{\lambda}{d_i}\right) + .84 \left(\frac{\lambda}{d_i}\right) e^{-(.44)(d/\lambda)} \right]$$

for T in °R, μ in poise, λ and d in cm.

User Instructions. Enter the program shown in Table D2.

To calculate μ and λ for Standard Air having 250°R ≤ T ≤ 600°R:

1. Load storage registers with the following variables

| | | | |
|---|---|---|---|
| Temperature | T, | °R, | STO 2 |
| Absolute Pressure, | P, | "Hg, | STO 3 |

2. Start program "μ, λ" "B"

3. Output:

Values of μ are stored in the correct storage register for retrieval during calculation of D

| | |
|---|---|
| To display μ (units of poise) | RCL 4 |
| To display λ (units of cm) | RCL 5 |

To calculate $D_i$ for a monodisperse aerosol

4. Load storage registers with the following variables

| | | | | |
|---|---|---|---|---|
| Temperature, | T, | R, | STO 2 | Performed |
| Viscosity, | μ, | poise, | STO 4 | in Steps |

## TABLE D2

## PROGRAM 1 - PARTICLE DIFFUSIVITY

## HP-65 CALCULATOR PROGRAM FOR A PARALLEL PLATE DIFFUSION BATTERY

| CODE | KEYS |
|---|---|
| LBL | 23 |
| A | 11 |
| RCL 1 | 34 01 |
| RCL 5 | 34 05 |
| ÷ | 81 |
| . | 83 |
| 4 | 04 |
| 3 | 03 |
| 5 | 05 |
| CHS | 42 |
| × | 71 |
| f⁻¹ | 32 |
| LN | 07 |
| RCL 5 | 34 05 |
| RCL 1 | 34 01 |
| ÷ | 81 |
| STO 8 | 33 08 |
| × | 71 |
| . | 83 |
| 9 | 09 |
| 4 | 04 |
| × | 71 |
| 1 | 01 |
| ÷ | 81 |
| RCL 8 | 34 08 |
| 2 | 02 |
| . | 83 |
| 4 | 04 |
| 0 | 00 |
| 2 | 02 |
| × | 71 |
| ÷ | 81 |
| RCL 2 | 34 02 |
| × | 71 |
| 1 | 01 |
| 6 | 06 |

| CODE | KEYS |
|---|---|
| 6 | 06 |
| EEX | 43 |
| CHS | 42 |
| 1 | 01 |
| 9 | 09 |
| × | 71 |
| RCL 4 | 34 04 |
| ÷ | 81 |
| RCL 1 | 34 01 |
| ÷ | 81 |
| STO 7 | 33 07 |
| RTN | 24 |
| LBL | 23 |
| B | 12 |
| . | 83 |
| 4 | 04 |
| 0 | 00 |
| 5 | 05 |
| RCL 2 | 34 02 |
| 2 | 02 |
| 0 | 00 |
| 4 | 04 |
| − | 51 |
| × | 71 |
| 1 | 01 |
| 8 | 08 |
| 2 | 02 |
| + | 61 |
| EEX | 43 |
| CHS | 42 |
| 6 | 06 |
| × | 71 |
| STO 4 | 33 04 |
| RCL 3 | 34 03 |
| ÷ | 81 |
| RCL 2 | 34 02 |

| CODE | KEYS |
|---|---|
| f | 31 |
| ✓ | 09 |
| × | 71 |
| . | 83 |
| 0 | 00 |
| 5 | 05 |
| 7 | 07 |
| 7 | 07 |
| × | 71 |
| STO 7 | 33 07 |
| RTN | 24 |

| | | | | | |
|---|---|---|---|---|---|
| $R_1$ | d | $R_4$ | $\mu$ | $R_7$ | P |
| $R_2$ | T | $R_5$ | t | $R_8$ | (WORK) |
| $R_3$ | V | $R_6$ | (BLANK) | $R_9$ | (BLANK) |

| | | | | |
|---|---|---|---|---|
| Mean Free Path, | λ, | cm, | STO 5 | 1 and 2 |
| Diameter of the monodisperse aerosol, | $d_i$, | cm, | STO 1 | |

5. Start program "Diff" "A"

6. Output:

Displayed value is "D" in cgs units (also stored in register 7).

Test Problem

Find μ, λ, and $D_i$ for T = 75.2°F, P = 14.39 PSIA, and d = .023 microns.

1. Convert T, P, and $d_i$ to the proper units then store in registers 2, 3, and 1, respectively.

75.2°F = 24°C = 297°K, store in register 2

14.39 PSIA x 2.036 $\frac{\text{"Hg}}{\text{PSIA}}$ = 29.30 "Hg, store in register 3

.023 μm = .023 x $10^{-4}$ cm = 2.3 x $10^{-6}$ cm, store in register 1

2. Start Program B. λ is displayed (λ = 6.23 x $10^{-6}$ cm), RCL 4 to display (μ = 1.83 x $10^{-4}$ poise).

3. Start Program A. $D_i$ is displayed ($D_i$ = 9.95 x $10^{-5}$ cm²/sec).

## 2. Theoretical Penetration (Program Steps are Listed in Table D3.)

When the diffusivity ($D_i$) of a monodisperse aerosol of diameter $d_i$ is known, and the flow rate-geometry configuraion ($Y_j$) for the diffusional apparatus (diffusion battery) are known, fractional penetration as a function of size ($D_i$) is given by

$$P_{i,j} = 0.9104e^{-1.8852D_iY_j} + 0.0531e^{-21.431D_iY_j} + 0.0531e^{-62.317D_iY_j} + 0.068e^{-124.537D_iY_j}$$

If several diffusional configurations are used in series where the aerosol is allowed to remix before going through the next configuration, the final penetration is the product of the penetration for each, thus

$$P_i = \prod_j P_{i,j}$$

If m diffusion batteries having the same $Y_j$ value are used in series, then

$$P_i = (P_{i,j})^m$$

To calculate $P_{i,j}$ for m identical batteries in series, enter program in Table D3.

# TABLE 113

## PROGRAM 2 - PARTICLE PENETRATION

## HP-65 CALCULATOR PROGRAM FOR A PARALLEL PLATE DIFFUSION BATTERY

| CODE | KEYS |
|---|---|
| LBL | 23 |
| A | 11 |
| RCL 7 | 34 07 |
| RCL 8 | 34 08 |
| × | 71 |
| STO 8 | 33 08 |
| 1 | 01 |
| . | 83 |
| 9 | 09 |
| 8 | 08 |
| 5 | 05 |
| 2 | 02 |
| CHS | 42 |
| × | 71 |
| $f^{-1}$ | 32 |
| LN | 07 |
| . | 83 |
| 0 | 00 |
| 1 | 01 |
| 0 | 00 |
| 4 | 04 |
| × | 71 |
| RCL 8 | 34 08 |
| 2 | 02 |
| 1 | 01 |
| . | 83 |
| 4 | 04 |
| 3 | 03 |
| 1 | 01 |
| CHS | 42 |
| × | 71 |
| $f^{-1}$ | 32 |
| LN | 07 |
| . | 83 |
| 0 | 00 |
| 6 | 06 |

| CODE | KEYS |
|---|---|
| 3 | 03 |
| 1 | 01 |
| × | 71 |
| + | 61 |
| RCL 8 | 34 08 |
| 8 | 08 |
| 2 | 02 |
| . | 83 |
| 3 | 03 |
| 1 | 01 |
| 7 | 07 |
| CHS | 42 |
| × | 71 |
| $f^{-1}$ | 32 |
| LN | 07 |
| . | 83 |
| 0 | 00 |
| 1 | 01 |
| 5 | 05 |
| 3 | 03 |
| × | 71 |
| + | 61 |
| RCL 8 | 34 08 |
| 1 | 01 |
| 2 | 02 |
| 4 | 04 |
| . | 83 |
| 5 | 05 |
| 3 | 03 |
| 7 | 07 |
| CHS | 42 |
| × | 71 |
| $f^{-1}$ | 32 |
| LN | 07 |
| . | 83 |
| 0 | 00 |

| CODE | KEYS |
|---|---|
| 0 | 00 |
| 6 | 06 |
| 8 | 08 |
| × | 71 |
| + | 61 |
| RCL | 34 |
| 9 | 09 |
| g | 35 |
| $y^x$ | 05 |
| RTN | 24 |

| | | | | | |
|---|---|---|---|---|---|
| $R_1$ | (BLANK) | $R_4$ | (BLANK) | $R_7$ | D |
| $R_2$ | (BLANK) | $R_5$ | (BLANK) | $R_8$ | (WORK) |
| $R_3$ | P | $R_6$ | V | $R_9$ | m |

1. Load storage registers with the following variables

| | | | |
|---|---|---|---|
| Diffusivity, | $D_i$, | cgs units, | STO 7 |
| Flow rate-geometry configuration | $V_j$, | cgs units, | STO 6 |
| Number of identical batteries in series | m, | integer, | STO 9 |

2. Start Program "A"

Displayed value is the fractional penetration ($P_{i,j}$) of this monodisperse aerosol of size $d_i$ through n identical diffusion batteries in series.

Test Problems

1. Load data

$V_j$ = 2.36 x $10^{3}$ in cgs units STO 6
$D_i$ = 5.39 x $10^{-6}$ in cgs units STO 7
$P_{i,j}$ = 1 STO 9

2. Calculate P "A"

P = 9.38 x $10^{-1}$ = 93.8%

Therefore the penetration of the diffusion battery Type A at a flow rate of 6 liters/minute is 93.8% for 0.023 μm particles.

After calculating the penetration, a graph similar to Figure D9 will be obtained. The $D_{50}$'s can be determined from this graph as indicated. The experimental data for this sample calculation are shown in Figure D11.

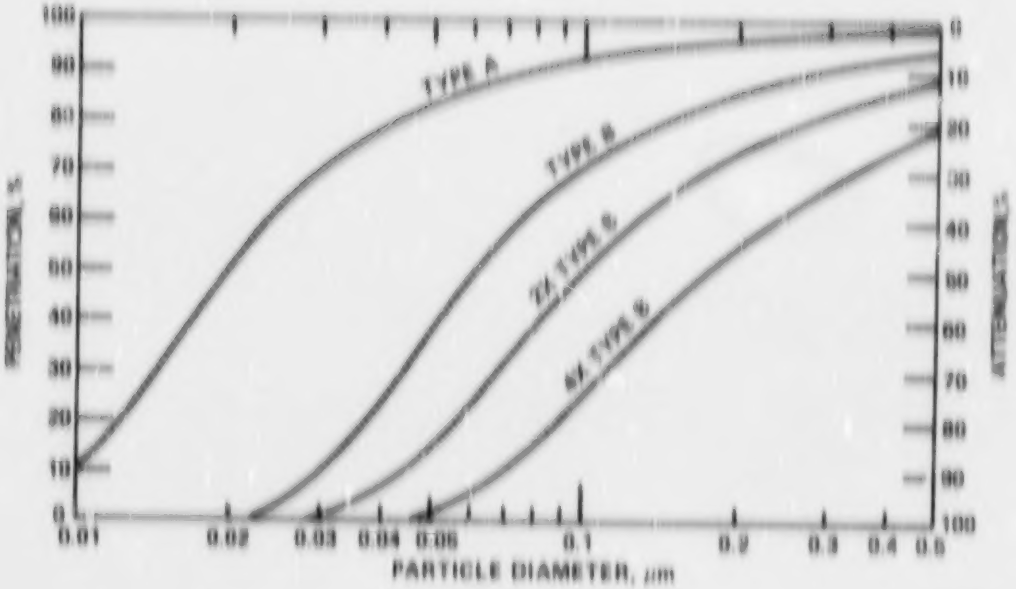

*Figure D.9. Theoretical parallel plate diffusion battery penetration curves.*

A cumulative particle size distribution is plotted using the corrected concentration from the last column in Table D1, as the ordinate and the $D_{50}$'s for each configuration as the abscissa. Differential number graphs are obtained by differentiation of the cumulative curve (finite differences) as described in Sections C.8 and C.9.

## D.3. SUBMICRON PARTICLE SIZING TECHNIQUES USING THE ELECTRICAL MOBILITY PRINCIPLE

### D.3.1 Electrical Mobility

If a particle charge is known, measurements of the electrical mobility are sufficient to determine the particle size. This concept has been used by Liu, *et al.*,[1] at the University of Minnesota to develop a series of Electrical Aerosol Analyzers (EAA). A schematic of this device is shown in Figure D10. The EAA has the distinct advantage of very rapid data acquisition compared to parallel plate diffusion batteries and condensation nuclei counters (two minutes as opposed to two hours for a single size distribution analysis.

### D.3.2 Thermosystems Model 3030 Electrical Aerosol Size Analyser*

The EAA is designed to size particles in the range of 0.01 micrometer diameter to 1.0 micrometer diameter. It can size solids and non-volatile liquids. The concentration of 1 to 1000 $\mu g/m^3$ limit its application to a sample extraction-dilution system as described earlier.

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*Thermosystems, Inc., St. Paul, MN 55113.

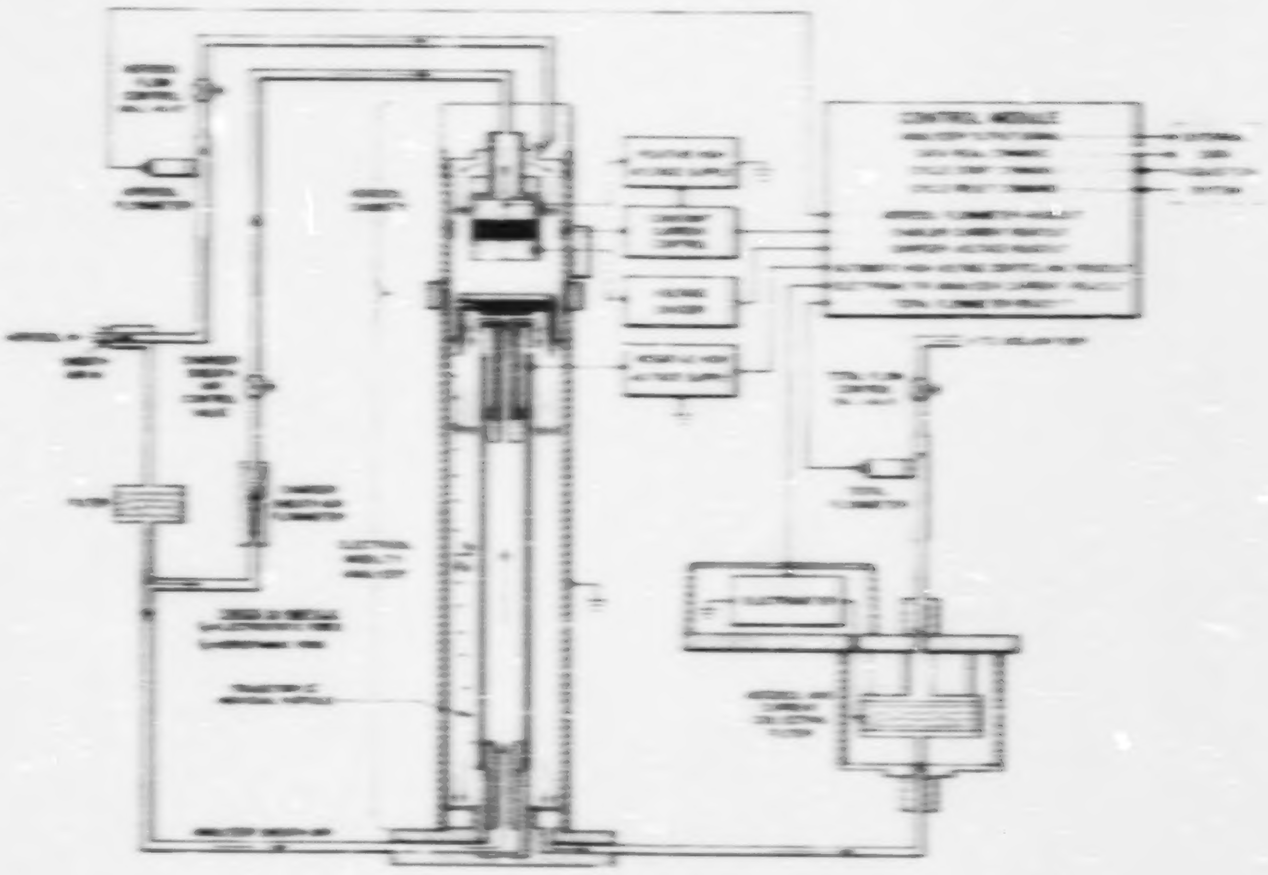
CONTROL MODULE

Figure D10 Schematic diagram of electrical aerosol analyser.
After Liu and Pui.

The EAA operated in the following manner. As a vacuum pump draws the aerosol through the analyzer (See Figure D10), a corona generated at a high voltage wire within the charging section gives the sample a positive electrical charge.

The charged aerosol flows from the charger to the analyzer section as an annular cylinder of aerosol surrounding a cone of clean air. A metal rod, to which a variable, negative voltage can be applied, passes axially through the center of the analyzer tube. Particles smaller than a certain size (with highest electrical mobility) are drawn to the collecting rod when the voltage corresponding to that size is on the rod. Larger particles pass through the analyzer tube and are collected by a filter. The electrical charges on these particles drain off through an electrometer, giving a measure of current.

A step increase in rod voltage will cause particles of a larger size to be collected by the rod with a resulting decrease in electrometer current. This decrease in current is related to the additional number of particles being collected. A total of eleven voltage steps divide the 0.0032 to 1.0 micron size range of the instrument into ten equal logarithmic size intervals. Different size intervals can be programmed via an optional plug-in memory card.

The electrical aerosol analyzer can be operated either automatically or manually. In the automatic mode, the analyzer steps through the entire size range. For size and concentration monitoring over an extended period of time, the analyzer may be intermittently triggered by an external timer. The standard readout consists of a digital display within the control circuit module, although a chart recorder output is available. It is almost always advantageous to use a strip chart recorder to record the data. This allows the operator to identify a stable reading superimposed on source variations and gives a permanent record of the raw data.

### D.3.3 Using The TSI Model 3030 Electrical Aerosol Analyzer With a Sample Extraction-Dilution System To Measure Concentrations of Submicron Aerosols in Industrial Flue Gases

Once the equipment is set up as shown in Figure D3, the flows are adjusted through the sample orifice and the dilution air orifice, to obtain the desired dilution factor. The EAA is placed in a manual mode and the current readings for each channel are recorded with a strip chart recorder. Manual control allows run times of from two to five minutes in each of the nine channels. This allows one to average out rapid source fluctuations. At the beginning of each day, the interval calibration points and flows through the EAA are checked, as described in the instrument manual. These are periodically rechecked throughout the day.

### D.3.4 Data Reduction Techniques and a Sample Calculation

It is assumed that a Thermo-Systems Inc. Model 3030 Electrical Aerosol Size Analyzer (EAA) with a 0.0032 μm to 0.360 μm range at the normal operating conditions has been used to determine concentration *vs* size information in the ultrafine size range for the effluent of a precipitator. The EAA sampled the gas stream after the sample was extracted with a Sample Extraction and Dilution System as described in Section D.1.7.

# TABLE D4

EAA (Model 3030) Data Reduction Form
Concentration, Cumulative Concentration, and $\Delta N_s/\Delta \log D$ from Scan No. ____
for DF = ____

| 1<br>Channel No. | 2<br>Collector Voltage | 3<br>$D_p$, µm | 4<br>$D_{pi}$, µm | 5<br>$\Delta N/\Delta I$ | 6<br>$\Delta \log D_p$ | 7<br>I, pA | 8<br>$\Delta I$, pA | 9<br>$\Delta N$ | 10<br>$\Delta N_s$ | 11<br>$\Sigma N_s$ | 12<br>$\Delta N_s/\Delta \log D$ |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 3 | 196 | 0.0100 | | | | ____ | | | | | |
| | | | 0.0133 | $4.76\times10^5$ | 0.253 | | ____ | ____ | ____ | ____ | ____ |
| 4 | 593 | 0.0179 | | | | ____ | | | | | |
| | | | 0.0215 | $2.33\times10^5$ | 0.162 | | ____ | ____ | ____ | ____ | ____ |
| 5 | 1220 | 0.026 | | | | ____ | | | | | |
| | | | 0.0306 | $1.47\times10^5$ | 0.141 | | ____ | ____ | ____ | ____ | ____ |
| 6 | 2183 | 0.036 | | | | ____ | | | | | |
| | | | 0.0502 | $8.33\times10^4$ | 0.289 | | ____ | ____ | ____ | ____ | ____ |
| 7 | 3515 | 0.070 | | | | ____ | | | | | |
| | | | 0.0917 | $4.26\times10^4$ | 0.234 | | ____ | ____ | ____ | ____ | ____ |
| 8 | 5387 | 0.120 | | | | ____ | | | | | |
| | | | 0.149 | $2.47\times10^4$ | 0.188 | | ____ | ____ | ____ | ____ | ____ |
| 9 | 7152 | 0.185 | | | | ____ | | | | | |
| | | | 0.219 | $1.56\times10^4$ | 0.148 | | ____ | ____ | ____ | ____ | ____ |
| 10 | 8642 | 0.260 | | | | ____ | | | | | |
| | | | 0.306 | $1.10\times10^4$ | 0.141 | | ____ | ____ | ____ | ____ | ____ |
| 11 | 9647 | 0.360 | | | | ____ | | | | | |

The EAA was placed in a manual scan mode and the current readings for each channel were recorded with a strip chart recorder. Manual control allowed run times of from two to five minutes in each of the nine channels. This allowed the averaging of rapid source fluctuations.

The theory of operation and basic equations for the EAA have been given by Liu *et al*[21] and calibration of the Model 3030 EAA has been done by Liu and Pui.[22] The latest calibration and modifications revise the initial EAA calibration which assumed eleven size fractions from .0032 μm to 1.0 μm, but which was actually eleven size fractions from 0.0032 μm to 0.36 μm (as usual in this example). The mechanics of the data reduction is identical, however. Table D4 shows the previous calibration constants in a data reduction format. The calibration by Liu[22] suggested the use of a calibration matrix; however, typical source fluctuations in industrial processes generally negate any potential advantage of such refinements. Table D4 is essentially self-explanatory. The heading "$D_p$, μm" (Column 3) is the particle diameter in micrometers. A value of 0.100 μm diameter and smaller are collected in the analyzer tube while larger particles penetrate to the current collecting filter where an electrometer measures the total current carried by the unprecipitated particles. This current represents the charges on all particles larger than 0.100 μm. This measured current is the basic output of the Model 3030.

The fourth column ($D_{pi}$, μm) is the geometric mean diameter of the particles represented by the current difference of two successive steps (Channel No.'s). For example, the difference in current for the 0.100 μm cut-off and the current for the 0.0178 μm cut-off is the total current collected from particles between these sizes, or rather for a mean diameter of 0.0133 μm. The current differences are entered in Column 8 headed " I, pA" (picoAmps).

B. Calculate all dilution factors ($DF_j$).

STEP 2

Calculate current differences ($\Delta I_{i,j}$) from adjacent channels and average the $\alpha_i$ products ($\alpha_i = \Delta I_{i,j} \times DF_j$) for the same size band for all scans taken for the same test conditions. Calculate 90% confidence intervals for each $\bar{\alpha}_i$. Note: the i subscript denotes size and the j subscript denotes dilution setting.

STEP 3

Using $\bar{\alpha}_i$ and Table D6 calculate "number concentration" ($\Delta N_s$), "average cumulative concentration of all particles having diameter greater than the indicated size" $\Sigma(\Delta N_s)$, and "$\Delta N_s/\Delta \mathrm{Log}D$" for each size band for each test condition.

STEP 4

Plot "Cumulative Concentration vs. Size" for each test condition.

STEP 5

Plot $\Delta N_s/\Delta \mathrm{Log}D$ (with upper and lower 90% confidence limits) vs. size for each test condition.

SAMPLE CALCULATION FOLLOWING THE CALCULATION FORMAT

Table D5 contains hypothetical test data for the following sample calculation.

# TABLE 25

SEM Current Readings (I, in picoamps) and Dilution Factors
for this Sample Calculation: Hypothetical Inlet Data

| SCAN | Time | CH 3 | CH 4 | CH 5 | CH 6 | CH 7 | CH 8 | CH 9 | CH 10 | CH 11 | Dilution Factor |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 1:30p | 2.089 | 2.734 | 2.529 | 2.227 | 1.362 | .482 | .242 | .102 | .030 | 255 |
| 2 | 1:32 | 2.055 | 2.711 | 2.485 | 2.205 | 1.344 | .469 | .220 | .075 | −.010 | 255 |
| 3 | 1:34 | 2.061 | 2.709 | 2.508 | 2.209 | 1.340 | .455 | .219 | .081 | .001 | 255 |
| 4 | 1:36 | 2.089 | 2.722 | 2.522 | 2.225 | 1.360 | .476 | .226 | .096 | .020 | 255 |
| 5 | 1:38 | 2.086 | 2.746 | 2.530 | 2.232 | 1.381 | .756 | .279 | .127 | .052 | 255 |
| 6 | 1:40 | 2.066 | 2.736 | 2.531 | 2.210 | 1.370 | .488 | .255 | .125 | .033 | 255 |
| 7 | 1:45 | 6.477 | 6.288 | 5.716 | 5.056 | 3.222 | 1.575 | .565 | .342 | .053 | 133 |
| 8 | 1:47 | 6.588 | 6.288 | 5.810 | 5.251 | 3.223 | 1.613 | .518 | .295 | .018 | 133 |
| 9 | 1:49 | 6.277 | 6.187 | 5.623 | 4.965 | 3.022 | 1.526 | .537 | .227 | .032 | 133 |
| 10 | 1:51 | 6.328 | 6.096 | 5.616 | 4.956 | 2.866 | 1.467 | .465 | .207 | .035 | 133 |

## TABLE 26

### EAA (Model 3030) Data Reduction Form
### Concentration, Cumulative Concentration, and $\Delta N_s/\Delta\log D$
### From Average $\bar{n}$ for Condition ______

| 1<br>Channel No. | 2<br>Collector Voltage | 3<br>$D_p$, um | 4<br>$D_{pi}$, um | 5<br>$\Delta N/\Delta I$ | 6<br>$\Delta\log D_p$ | 7<br>$\bar{n}$ | 8<br>$\Delta N_s$ | 9<br>$\Sigma\Delta N_s$ | 10<br>$\Delta N_s/\Delta\log D$ |
|---|---|---|---|---|---|---|---|---|---|
| 3 | 196 | 0.0100 | | | | | | | |
| | | | 0.0133 | $4.76\text{x}10^{-5}$ | 0.250 | ____ | ____ | ____ | ____ |
| 4 | 593 | 0.0178 | | | | | | | |
| | | | 0.0215 | $2.33\text{x}10^{-5}$ | 0.165 | ____ | ____ | ____ | ____ |
| 5 | 1220 | 0.026 | | | | | | | |
| | | | 0.0306 | $1.47\text{x}10^{-5}$ | 0.141 | ____ | ____ | ____ | ____ |
| 6 | 2183 | 0.036 | | | | | | | |
| | | | 0.0502 | $8.33\text{x}10^{-6}$ | 0.289 | ____ | ____ | ____ | ____ |
| 7 | 3515 | 0.070 | | | | | | | |
| | | | 0.0917 | $4.26\text{x}10^{-6}$ | 0.234 | ____ | ____ | ____ | ____ |
| 8 | 5387 | 0.120 | | | | | | | |
| | | | 0.149 | $2.47\text{x}10^{-6}$ | 0.188 | ____ | ____ | ____ | ____ |
| 9 | 7152 | 0.185 | | | | | | | |
| | | | 0.219 | $1.56\text{x}10^{-6}$ | 0.148 | ____ | ____ | ____ | ____ |
| 10 | 8642 | 0.260 | | | | | | | |
| | | | 0.306 | $1.10\text{x}10^{-6}$ | 0.141 | ____ | ____ | ____ | ____ |
| 11 | 9647 | 0.360 | | | | | | | |

The fifth column gives the revised calibration factor (based on the calibration by Liu and Pui[11]) for each of the eight size bands. These factors are in units of particles per cm per picoAmpere. Multiplying this size specific current sensitivity, $\Delta N/\Delta I$, (Column 5) by the current difference, $\Delta I$, (Column 8) gives the total number of particles, $\Delta N$, (Column 9) in units of particles per $cm^3$, within this size band (Column 4) for the diluted aerosol. To correct for dilution and find in-stack concentrations, multiply Column 9 by the dilution factor (DF) and enter the result, $\Delta N_s$, in Column 10. Columns 6 and 12 are used for $\Delta N_s/\Delta \mathrm{LogD}$ information calculated from the number distribution in Column 10. Column 11 is used for cumulative concentrations, corrected for dilution to engineering standard (normal) conditions by a dilution factor (i.e. Column 10). Engineering standard or normal conditions are defined as dry gas at 21°C and 760 mm Hg pressure.

The basic data from the EAA is cumulative current for each of nine channels (Column 7). One must then take the differences of the current readings for successive channels (Column 8) in order to find $\Delta N$, etc. These I values are multiplied by a series of constants $(\Delta N/\Delta I_i, DF_j)$ to arrive at $\Delta N_s$ (concentration in stack corrected to dry, standard conditions). While a single scan should be made at a constant dilution, different scans may be made at different dilutions. To simplify the aritnmetic for each test condition, the product $\alpha_i = \Delta I_{i,j} \times DF_j$ is formed and all such inlet (outlet) products for the same size band are averaged.

## SUMMARY OF THE CALCULATION FORMAT

### STEP 1

A. Calculate the average instrument reading (I) for each channel as obtained from the strip chart recording of channel current vs. time.

STEP 1

A. Calculate the average instrument reading (I) for each channel as obtained from the strip chart recording of channel current vs. time. Each complete size scan (Table D5) consists of nine instrument readings (I, Column 7 of Table D4). These instrument readings are the average current outputs as taken from the strip chart recordings, for each of the nine channels. Run times were manually controlled and varied from two to ten minutes per channel as the instrument operator sequentially stepped through channels 3, 4, 5, ..., 11. Table D5 gives the instrument readings used as data for the sample calculation (10 scans, 90 average current readings).

B. Calculate all dilution factors ($DF_j$) corrected to engineering standard (normal) conditions: 70°F (20°C) and 29.92 inches of mercury pressure (760 mm Hg)).

STEP 2

Calculate current differences ($\Delta I_{i,j}$) from adjacent channels and average the $\alpha_i$ products for the same size band for all scans taken at the same test condition. Calculate 90% confidence intervals for each $\bar{\alpha}_i$. (Refer to Section C.8.5).

The $\alpha_i$ product is given by the following:

$$\alpha_i = \Delta I_{i,j} \times DF_j$$

where i denotes the size band and j denotes the dilution value.

For channels 3-4 we have:

Scan #1: $\alpha_{3-4,1}$ = (.135)(255) pA

#2: $\alpha_{3-4,1}$ = (.124)(255) pA

#3: $\alpha_{3-4,1}$ = (.132)(255) pA

⋮

$\#9: \quad \bar{I}_{3-4,2} = (.290)(113)$ pA

$\#10: \quad \bar{I}_{3-4,2} = (.296)(113)$ pA

thus $\bar{I}_{3-4} = 33.179$ pA; n = 10 and CI = .579.

In a similar manner we can find $\bar{I}_{4-5}, \bar{I}_{5-6}, \ldots, \bar{I}_{10-11}$.

Thus the mean, with upper and lower 90% confidence limits for $\bar{I}_{3-4}$ is given by:

$$\bar{I}_{3-4} = (33.179 \pm 0.579) \text{ pA}$$

or

$$\bar{I}_{3-4} = (33.2 \pm 0.6) \text{ pA}$$

STEP 3

Using $\bar{I}_i$ and Table D6 calculate "number concentration" ($\Delta N_B$), "average cumulative concentration . . ." ($\Sigma\Delta N_B$), and "$\Delta N_B/\Delta$LogD" for each size band for each test condition.

Table D7 shows these calculations for the sample data of Table D5. Column 7 is $\bar{I}$ as shown in Step 2. Column 8 is the product of columns 7 and 5. Column 9 is the summation of 8 for all sizes "equal to or greater than the indicated size." Column 10 is column 5 times column 7 divided by column 6.

STEP 4

Plot cumulative concentration vs. size for each test conditions. For the sample data set of Table D5 this would be the concentrations in Table D7 column 9 plotted against the sizes in column 4. No errors bars are used.

## TABLE 37

### EAA (Model 3030) Data Reduction Form
### Concentration, Cumulative Concentration, and $\Delta N_s/\Delta \log D$
### from Average ΔI for Condition Inlet
### (Sample Calculation)

| 1<br>Channel No. | 2<br>Collector Voltage | 3<br>$D_p$, µm | 4<br>$D_{pi}$, µm | 5<br>ΔN/ΔI | 6<br>$\Delta \log D_p$ | 7<br>$\bar{I}$ | 8<br>$\Delta N_s$ | 9<br>$\Sigma N_s$ | 10<br>$\Delta N_s/\Delta \log D$ |
|---|---|---|---|---|---|---|---|---|---|
| 3 | 196 | 0.0100 | | | | | $\times 10^6$ | $\times 10^6$ | $\times 10^6$ |
| 4 | 593 | 0.0178 | 0.0133 | $4.76\times10^5$ | 0.250 | 33.2±.6 | 25.8±.3 | 68.4 | 63.2±1.1 |
| 5 | 1220 | 0.026 | 0.0215 | $2.33\times10^5$ | 0.165 | 53.3±.7 | 12.4±.2 | 52.6 | 75.3±1.0 |
| 6 | 2183 | 0.036 | 0.0306 | $1.47\times10^5$ | 0.141 | 74.3±.8 | 10.9±.1 | 40.2 | 77.5±.8 |
| 7 | 3515 | 0.070 | 0.0502 | $8.33\times10^4$ | 0.289 | 119.8±.8 | 10.3±.1 | 21.3 | 61.0±.2 |
| 8 | 5387 | 0.128 | 0.0917 | $4.26\times10^4$ | 0.234 | 174±2 | 7.42±.09 | 11.0 | 31.7±.4 |
| 9 | 7151 | 0.185 | 0.149 | $2.47\times10^4$ | 0.188 | 114±2 | 2.82±.05 | 3.61 | 15.2±.3 |
| 10 | 8642 | 0.260 | 0.219 | $1.56\times10^4$ | 0.148 | 35.4±.6 | .552±.009 | .785 | 3.73±.06 |
| 11 | 9647 | 0.360 | 0.306 | $1.10\times10^4$ | 0.141 | 21.2±.3 | .233±.003 | .233 | 1.65±.02 |

STEP 5

Plot $\Delta N_s/\Delta \text{Log} D$ with upper and lower 90% confidence limits for each test condition.

For the sample data set of Table D5 this would be the concentrations in Table D7, column 10 plotted against the sizes in column 4. The upper error bar is the value plus the 90% confidence interval. The lower error bar is the value minus the 90% confidence interval. For $\bar{N}_{3-4}$ in Table D7 we would have $\bar{N}_{3-4} = 33.2 \pm 0.6$
thus:

$$\Delta N_s/\Delta \text{LogD} = \frac{33.2 \times 4.76 \times 10}{.250} \pm \frac{0.6 \times 4.76 \times 10}{.250}$$

$$= (63.2 \pm 1.1) \times 10^{2}$$

The data shown in Column 10, Table D7 is graphically displayed in Figure D-11.

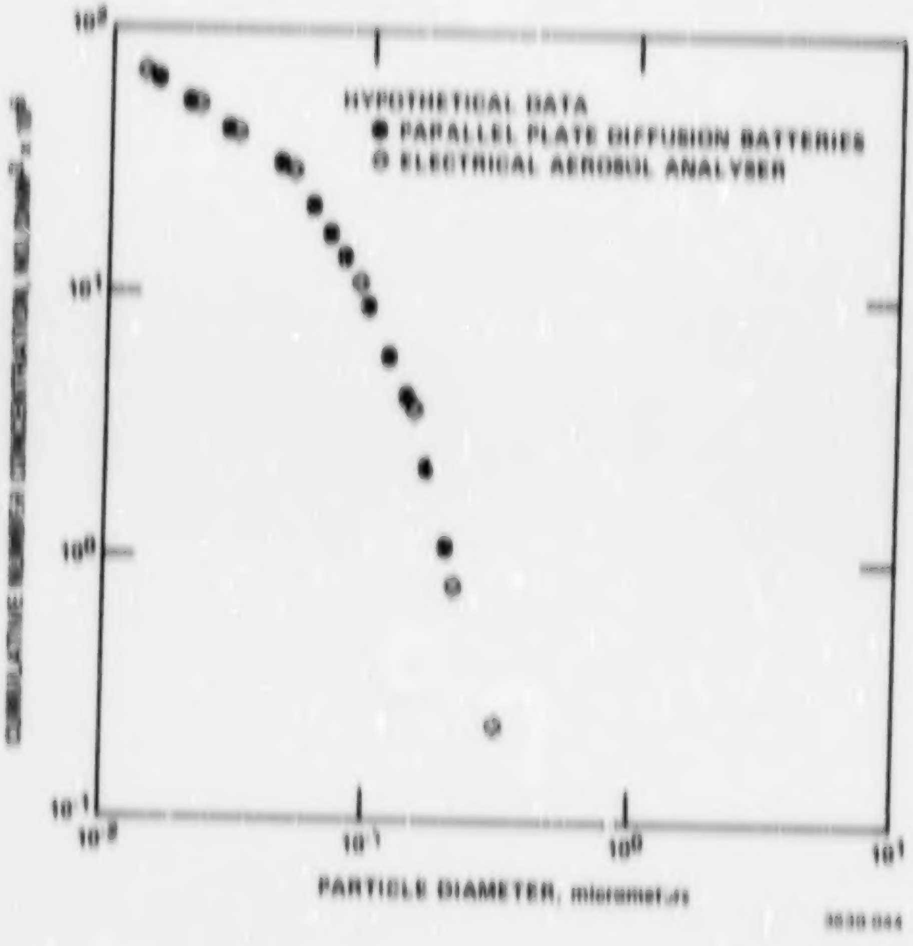

*Figure D11. Hypothetical inlet size distribution at an ESP on a coal-fired boiler. Sample data for parallel plate diffusion batteries and electrical aerosol analyser are shown.*

]

## D.4 OPTICAL PARTICLE COUNTERS

Optical particle counters are a useful adjunct to the ultrafine sizing system, giving additional data in the region where impactors, diffusional and electrical methods have the worst resolution.

A number of commercial optical particle counters are available which will perform adequately in the field environment associated with precipitator evaluation. These instruments can be obtained with several optical sensor configurations and may usually be specified to perform in a threshold mode, where all particles larger than a certain size are counted, or in a window mode where a narrow range of particle sizes is indicated. In either instance, the smallest particle which will be detected is approximately 0.3 μm diameter. In this respect, most optical counters size particles in the same range as cascade impactors and cyclones.

The calibration of optical particle counters is usually done with monodisperse plastic spheres which have a refractive index of 1.59. Any deviations from this index of refraction, or sphericity of the particles will cause the indicated sizes to be in error. If high accuracy in the optical particle counter data is important to the test, it is possible to do calibrations in the field, using the test aerosol. McCain[5] has used a sedimentation technique which employs parallel plate diffusion batteries as sedimentation chambers to correlate aerodynamic particle diameter with the indicated, or equivalent PSL, diameter. Marple[41] reported the development of special impaction devices which may be used for this same purpose.

In the majority of full sampling situations, optical particle counters must be used with some type of Sample Extraction and Dilution System as described in Section D.1. The sample is thus cooled and diluted to less than 300 particles per $cm^3$. Also it is usually necessary to place the return line back into the dilution system to minimize pressure gradients across the counter system.

Most commercially available systems come with real-time analog outputs and digital outputs with one to 10 minute accumulation or integration times. Analog signals are particularly useful for monitoring source variations and rapping emissions.

Data accumulated by optical particle counters are given as particles per unit volume and, after multiplication by the proper dilution factor, may be plotted on a cumulative or differential number basis. See paragraph C.8 for a discussion of data plotting. Figure 35 in Section 2.3.4. shows data taken using an optical-diffusional impactor system.

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# APPENDIX E

# LABORATORY DETERMINATION OF PARTICULATE RESISTIVITY

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  E.1.2 Procedure for Laboratory Resistivity Measurements
  E.1.3 Calculations

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# APPENDIX E

# LABORATORY DETERMINATION OF PARTICULATE RESISTIVITY

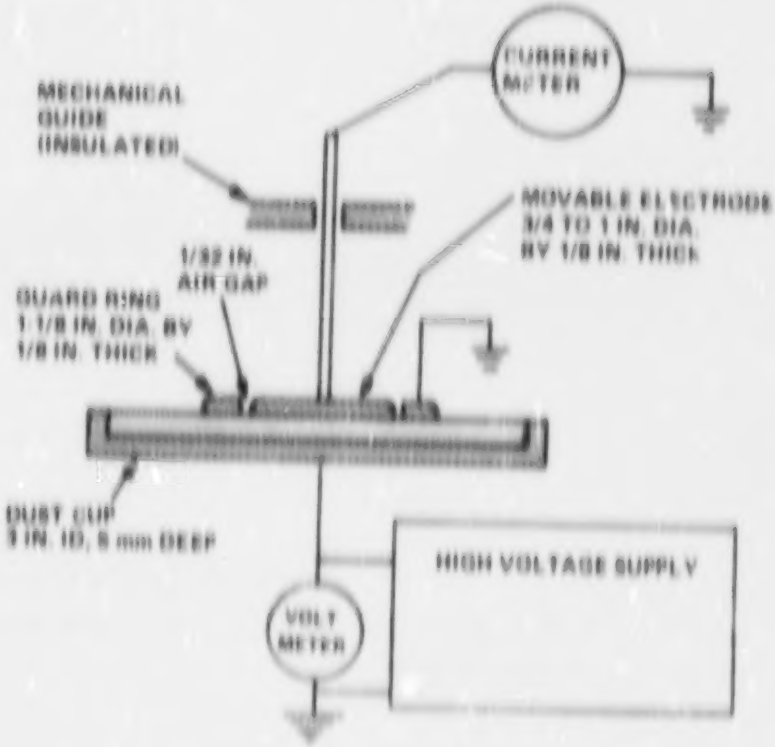
## E.1 STANDARD LABORATORY TECHNIQUE

The standard technique for conducting laboratory resistivity measurements is described in the American Society of Mechanical Engineers Power Test Code 28, Determining the Properties of Fine Particulate Matter.[1] This code was adopted by the Society in 1965 as a standard practice for the determination of all the properties of fine particulate matter which are involved in the design and evaluation of dust-separating apparatus. The tests include such properties as terminal settling velocity distribution, particle size, bulk electrical resistivity, water-soluble sulfate content, bulk density, and specific surface.

The document defines bulk electrical resistivity as the resistance to current flow, expressed in ohm-centimeters, through a dust sample contained in a cubic volume one centimeter on a side when exposed to an electrical voltage equivalent to 90% of the breakdown voltage of the sample, applied uniformly across two opposite faces of the cube. The code specifies that the property is to be determined at 150°C (300°F) and at a humidity of 5% by volume, unless otherwise specified.

### E.1.1 Apparatus

The basic resistivity cell is shown in Figure E1. It consists of a cup which contains the ash sample and serves as the lower electrode, and an upper electrode with a guard


CURRENT METER
MECHANICAL GUIDE (INSULATED)
MOVABLE ELECTRODE 3/4 TO 1 IN. DIA. BY 1/8 IN. THICK
1/32 IN. AIR GAP
GUARD RING 1 1/8 IN. DIA. BY 1/8 IN. THICK
DUST CUP 3 IN. ID, 5 mm DEEP
HIGH VOLTAGE SUPPLY
VOLT METER

0100 14 22

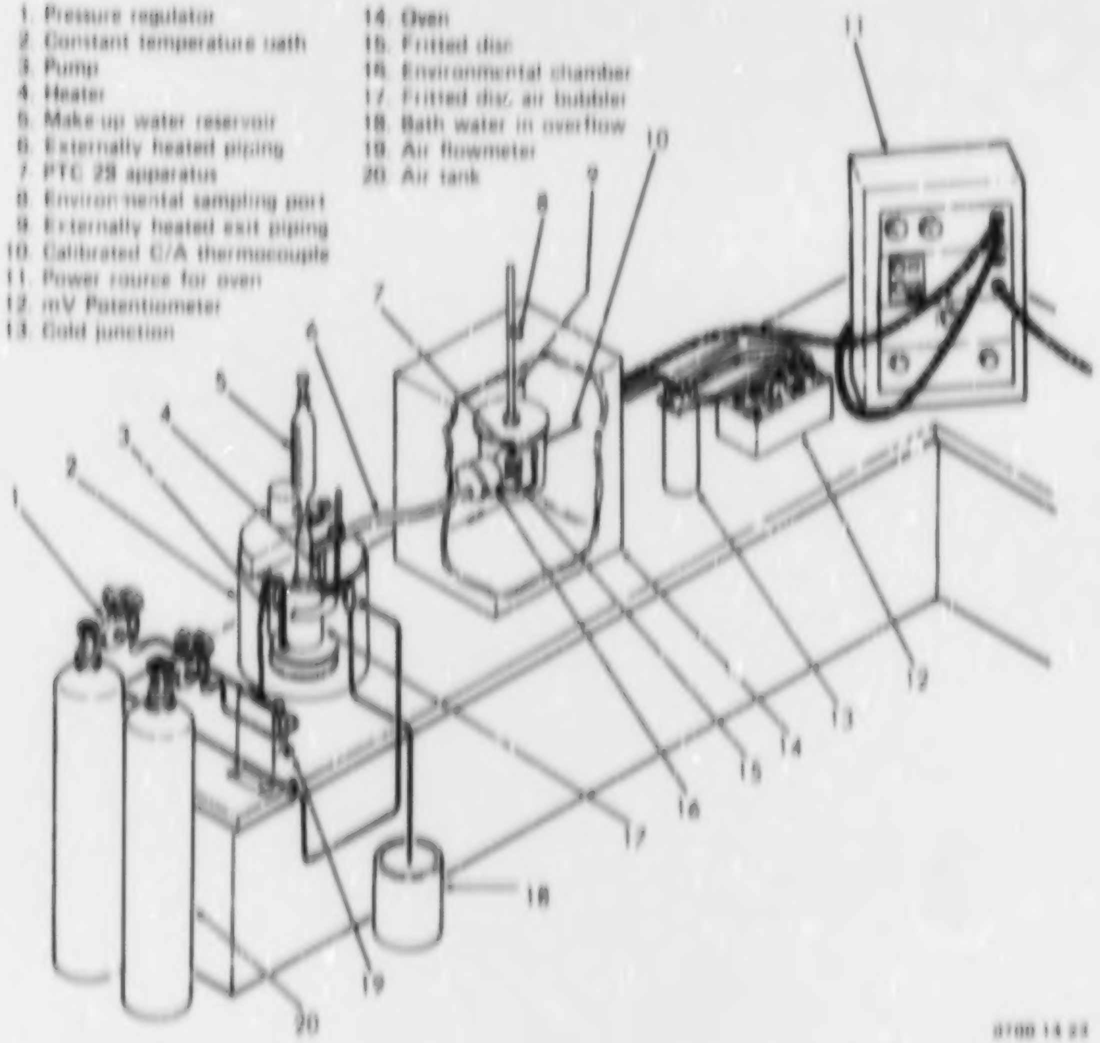
*Figure E1. Resistivity cell for laboratory measurements.*

ring. To conform with the code, the high-voltage resistivity cell must have the dimensions shown, and must use electrodes constructed from 25-micron porosity sintered stainless steel.

The controlled environmental conditions required for the measurement of resistivity in the laboratory can be achieved by an electric oven with thermostatic temperature control and with good thermal insulation to maintain uniform internal temperature, and a means to control humidity. Humidity may be controlled by any one of several conventional means, including circulation of pre-conditioned gas through the oven, injection of a controlled amount of steam, use of a temperature-controlled circulating water bath, or the use of chemical solutions which control water vapor pressure. Figure E2 illustrates a suitable setup for resistivity measurements.

## E.1.2 Procedure for Laboratory Resistivity Measurements

The first problem encountered in making any resistivity measurement is obtaining an appropriate dust sample. The prescribed procedure for PTC 28 Code assumes that samples of gas-borne dust are taken from a duct in accordance with the Test Code For Determining Dust Concentration in a Gas Stream (PTC 27-1957).[3] The PTC 27 Code involves isokinetic dust sampling at various points in the duct. It is recommended that samples should not be obtained from a large bulk of material in a hopper, silo, or similar location. If it is necessary that samples be obtained from such a location, procedures which will insure that the sample is representative of the whole must be used. For any resistivity test to be performed on a bulk sample, it is necessary that a random sample be obtained. This can be done by quartering the bulk sample to


1. Pressure regulator
2. Constant temperature bath
3. Pump
4. Heater
5. Make-up water reservoir
6. Externally heated piping
7. PTC 28 apparatus
8. Environmental sampling port
9. Externally heated exit piping
10. Calibrated C/A thermocouple
11. Power source for oven
12. mV Potentiometer
13. Cold junction
14. Oven
15. Fritted disc
16. Environmental chamber
17. Fritted disc air bubbler
18. Bath water in overflow
19. Air flowmeter
20. Air tank
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0700 14 23

*Figure E2. Laboratory apparatus for measuring particulate bulk resistivity.*

obtain the test sample. To break up agglomerates and to remove foreign matter, e.g., collection plate scale, the specimen can be passed through an 80-mesh screen.

The procedure for making the resistivity measurement according to Power Test Code 28 follows:

1. The sample is placed in the cup of the resistivity cell by means of a spatula. Then it is leveled by drawing a straight edge blade held vertically, across the top of the cup.

2. The disc electrode is gently lowered onto the surface. It should rest freely on the sample surface without binding on any supports.

3. The resistivity cell is mounted in the environmental chamber and equilibrium temperature and humidity are established. The Code specifies that a temperature of 150°C (300°F) and a humidity of 5% by volume are to be used for the test, unless otherwise specified.

4. A low voltage is applied to the cell and then gradually raised in a series of steps up to the point of electrical breakdown of the sample layer. Current transients will occur when the voltage is first applied or increased across the cell. It is necessary that these die away before recording current and voltage readings (approximately one minute). A record of the current-voltage characteristic of the dust is obtained. Preferably using another sample, the above is repeated; when another sample is not available, the sample layer should be remixed and releveled after each run in order to break up any spark channels that may have been formed in the dust layer. A total of three runs should be made. The average breakdown

voltage is then calculated. Before taking the samples to breakdown, it is necessary to determine whether the temperature and moisture content of the sample are in equilibrium with temperature and humidity of the controlled environment. A test for equilibrium is that the voltage-current measurements are reproducible to within 10% when determined by two successive measurements made 15 minutes apart.

5. The resistivity of the samples is then calculated in the range of 85 to 95% of the average breakdown voltage, using the corresponding currents from the previously recorded voltage-current characteristics.

6. In any case, resistivity measurements at elevated temperatures and controlled humidity should be made under equilibrium temperature and moisture conditions between the gas and the dust. The condition for equilibrium of temperature and moisture conditions in the ash shall be determined by the requirement that resistivity measurements be reproducible within 10% when determined by two successive measurements 15 minutes apart. Further, since the particle conductivity usually depends on the past treatment of the particles, as well as on the temperature and humidity equilibrium reached under given conditions, it is preferable to determine the conductivity by raising the oven temperature to the value desired rather than by first heating above the desired temperature and then cooling to the desired temperature. For additional information, see Reference 3.

### B.1.3 Calculations

Resistivity can be calculated in the following way. First, calculate the resistance of the dust layer, R.

$$R(\text{ohms}) = \frac{V(\text{volts})}{I(\text{amps})} \qquad (B1)$$

Then calculate the resistivity, ρ.

$$\rho\,(\text{ohm-cm}) = R(\text{ohms})\,\frac{A(\text{cm}^2)}{\ell\,(\text{cm})} \qquad \text{(E2)}$$

The moisture content of the air in the environmental chamber can be determined by weighing a tube filled with calcium sulfate (Drierite) before and after passage of a measured volume of air through it. The volume of dry air passed through the tube is determined from the flow rate and the sampling time.

## E.2 An Alternate Laboratory Technique

An example[illegible] of an alternate laboratory technique is given below. This procedure has been quite useful for the comparison of resistivities for a large number of fly ashes over an extended temperature range. All procedural steps, equipment, calculations, etc. are identical to those stated in Section E.1. unless otherwise specified.

1. The resistivity cell and ash specimen are thermally equilibrated at 460°C overnight in an environment of dry air or nitrogen.

2. After determining the resistivity under condition 1 (usually a voltage of 2 kV/cm is used), the test environment is introduced. The environment can be an air-water mixture or a simulated flue gas without sulfur oxides. The voltage gradient and water concentration are selected with respect to the specific test objectives.

3. After the specimen "equilibrates" with the test environment, resistivity is determined, and the oven is turned off and allowed to cool naturally, 50°C to 100°C/hour.

4. As the test specimen cools, resistivity is determined at selected temperature intervals between 460°C and 85°C.

Figure E3 shows data acquired using this procedure.

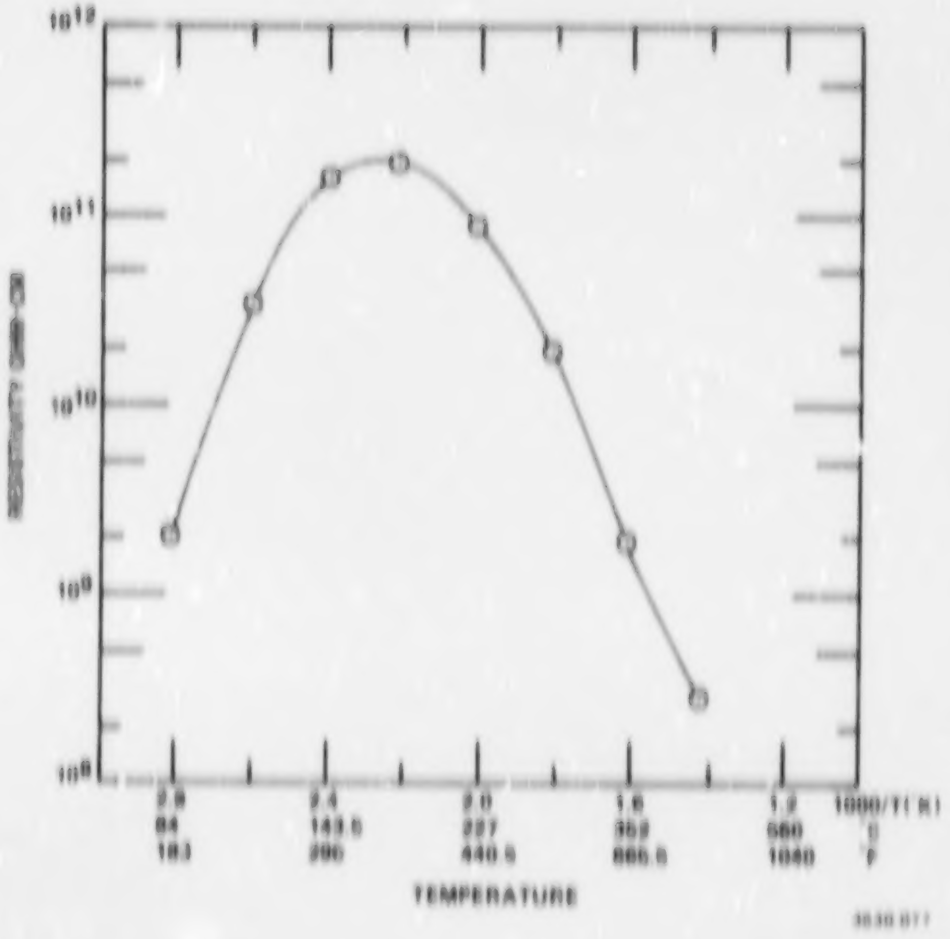

*Figure E.3. Resistivity versus temperature for fly ash obtained using the procedure outlined in Section E.2.*

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# APPENDIX F

## *IN SITU* PARTICULATE RESISTIVITY MEASUREMENTS

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## APPENDIX F

## IN SITU PARTICULATE RESISTIVITY MEASUREMENTS

Point-to-plane probes for measuring particle resistivity have been used since the early 1940's in this country.[1] Two models of these devices are shown in Figure F1.

This section describes the use of a particular point-to-plane resistivity probe. Similar procedures would be used with other point-to-plane resistivity probes. This probe can operate under the following conditions:

1. From 1 to 23 g/m$^3$ dust loadings (0.5 to 10 grains/ft$^3$).
2. Corrosive gas compositions.
3. Sampling ports of 10.2 cm to 15.4 cm diameter (4 inch to 6 inch pipe size).
4. Temperatures up to 200°C (392°F).
5. Resistivities of $10^5$ to $10^{13}$ Ω-cm.
6. Gas velocities of up to 30.5 m/sec (100 ft/sec).

This description is presented in the following sections: practical factors in resistivity measurement; a detailed description of the probe; general maintenance instructions; and operating instructions.

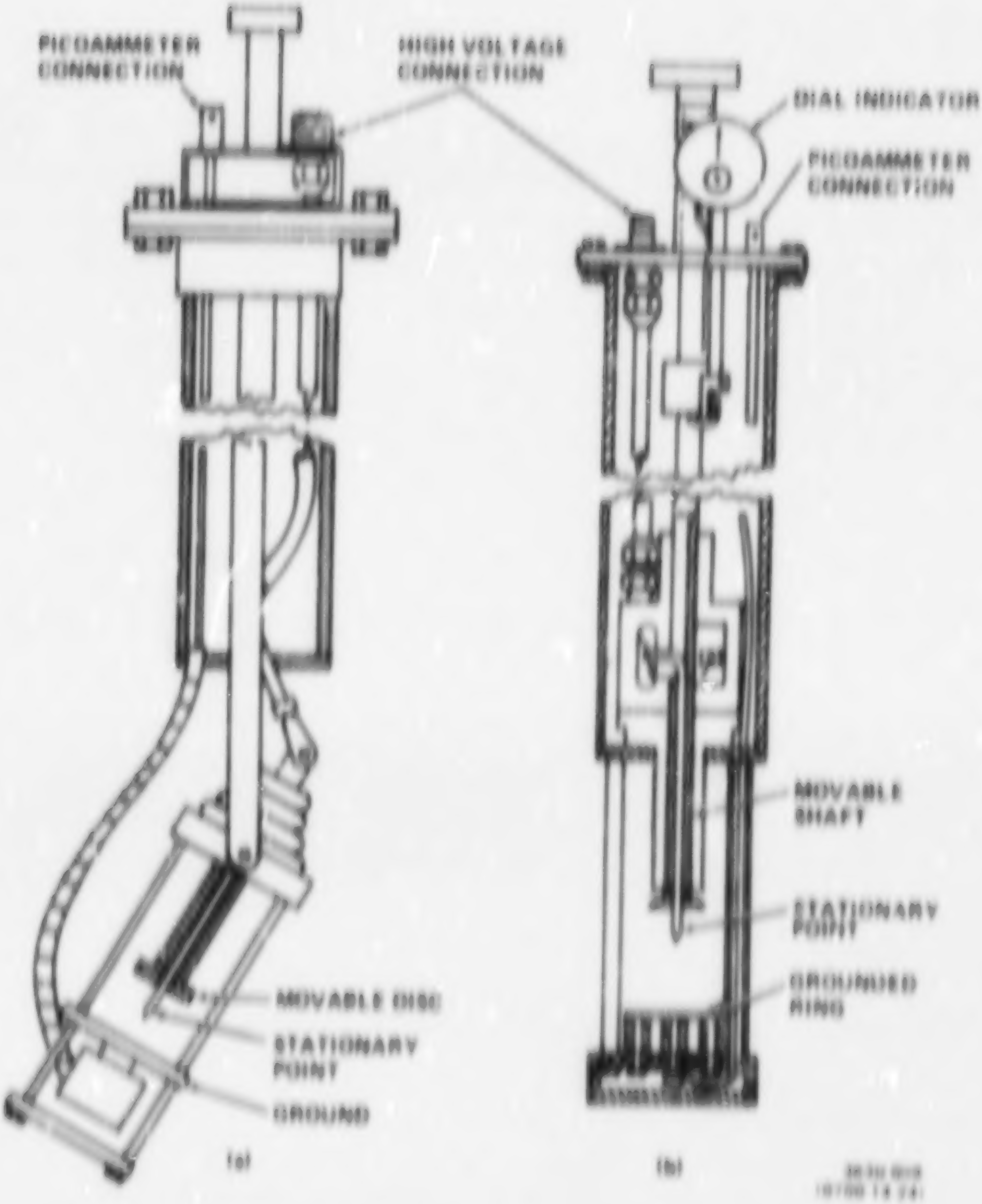
PICOAMMETER CONNECTION
HIGH VOLTAGE CONNECTION
DIAL INDICATOR
PICOAMMETER CONNECTION
MOVABLE SHAFT
STATIONARY POINT
GROUNDED RING
MOVABLE DISC
STATIONARY POINT
GROUND
(a)
(b)

*Figure F1. Two types of point-to-plane resistivity probes.*

Nichols[7] has written a more general description of methods for measuring particle resistivity, both laboratory and *in situ*.

## F.1 PRACTICAL FACTORS IN RESISTIVITY MEASUREMENTS

### F.1.1 Selection of Sampling Sites

The first priority in the selection of a sampling site is the location of a point in the operating system where the conditions of the gas and the gas-borne dust particles are representative of the environment for which the particle resistivity may be of practical significance. That is, the gas temperature, gas composition, and particle history should be the same as that found in the precipitator. Usually the inlet of the precipitator is selected as the point for making resistivity measurements. However, sampling at several points across the duct may be required to obtain a representative measurement if there are large variations in temperature across the duct.

When selecting a site for the measurements, practical considerations must also be remembered. At the site location, sampling ports must exist or be installed. The normal practice is to use 4-inch pipe for the ports. Electrical power must be available at the site location for the operation of the measuring equipment. In many locations, adapters will be required for mating of plant electrical outlets with the standard three-prong plugs found on most laboratory equipment.

### F.1.2 Number of Measurements Required

The determination of the number of individual measurements required to characterize the resistivity of the dust is related to the range of operating conditions anticipated and the variability in the process emissions. It is frequently desirable when gathering design data for a new precipitator installation, that the worst operating conditions be covered in the test schedule.

The variable plant operating condition that is of greatest concern is the flue gas temperature. Seasonal changes in the ambient air temperature can cause the flue gas temperature to fluctuate as much as 15°C (27°F) and the temperature profile across the duct downstream from a rotating air heater may show variations up to 25°C (45°F). Many cyclical processes have emissions with large fluctuations in particle and gas concentration, flowrate, and temperature. Changes in temperature may cause very large variations in the dust resistivity and care must be exercised to assure that the widest variation is anticipated and covered in the test plan.

The day-to-day variations in characteristics of the process or emissions may also cause significant variations in the particulate resistivity. This variability will show up as scatter in the measured value of resistivity over the measurement period. Thus it is imperative to make enough measurements at each operating condition to obtain a statistically significant value for the resistivity.

A precipitator acts to smooth out short-term variations in particulate resistivity. Dust layers ranging in thickness from perhaps one centimeter on the inlet plates to some lower value, perhaps only a millimeter, on the outlet plates build up during several hours of collection time. The average build-up rate on the precipitator plates is on the order of one millimeter per hour, exponentially distributed through the precipitator, so that the dust layer on the plates may represent an averaging of the instantaneous dust conditions for many hours of operation. Therefore, it is reasonable to average the measured values of resistivity for each temperature condition to arrive at the resistivity representative of the dust at a particular installation.

A determination of the number of measurement points required is based on the variability of the source and the experience of the technician making the mearurements. Typically, six to ten measurements, each at intervals of 10°C (18°F), are adequate if plant conditions are reasonably constant.

The auxiliary data required when conducting tests on an operating precipitator include:

Process material samples for proximate and ultimate analysis

Flue gas temperature and composition

Precipitator voltage-current relationships

Hopper dust samples for laboratory analysis.

## F.2 A POINT PLANE PROBE

System for making in situ resistivity measurements include a probe for insertion in the flue, a high voltage supply, a volt meter, an ammeter with overload protection, a temperature indicator, and a pump. A schematic diagram of a complete system is shown in Figure F2. A set of drawings showing the details of the design of a particular probe are shown in Figures

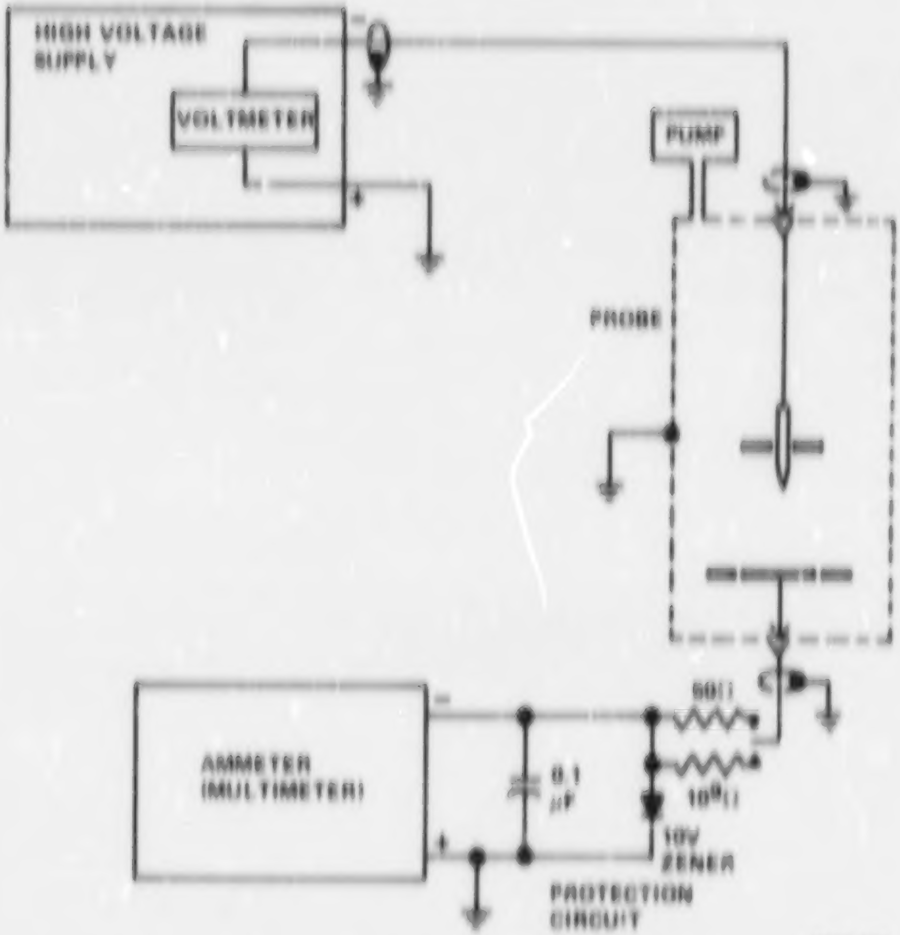
HIGH VOLTAGE SUPPLY
VOLTMETER
−
+
PUMP
PROBE
AMMETER (MULTIMETER)
−
+
0.1 μF
50Ω
10⁹Ω
10V ZENER
PROTECTION CIRCUIT

*Figure F2. Schematic diagram of probe system.*

F7-F13. The first drawing in this set gives an assembly view of the probe (drawing no. 2620-D-10).*

The power supply for the probe is a modified Peschel Model H20-Y (20 kVDC Neg. 4 mA) with two voltage scales (0-25 kV and 0-2.5 kV). The ammeter which is mounted in the power supply cabinet is a Keithley digital multimeter model 160 (sensitivity to currents as low as $10^{-11}$ amps). The input to the multimeter is protected from surge currents during sparkover by a zener diode protective circuit. This circuit also contains a 10 resistor for testing the probe.

The probe is equipped for collecting the dust, making electrical contact with the dust, measuring the thickness of the collected dust layer, and removing the collected dust layer, all without removing the probe from a sampling port.

The particulate sample is collected by a point plane corona discharge cell mounted in the end of the probe. The corona point (1203) is located 5.72 cm from the 5.2 um diameter collecting electrode (11A). The collecting electrode consists of a guard electrode (1108) and a center disc electrode (1104) (diameter 2.52 cm., area 5.0 $cm^2$). The guard electrode is

---

*Every item in the probe has a unique identification number consisting of the drawing number and the consecutive item number:

$$\underbrace{2620\text{-}D\text{-}17}_{\text{drawing no.}}\qquad\underbrace{01}_{\text{item}}$$

A reference to item 1703 means the third item of drawing 17. Every sub-assembly in the probe has a unique identification number consisting of the drawing number and an assembly letter:

$$\underbrace{2620\text{-}D\text{-}17}_{\text{drawing no.}}\qquad\underbrace{A}_{\text{assembly letter}}$$

All items in the material list bracketed with the assembly letter will be assembled into that sub-assembly. A reference to assembly 17A means sub-assembly A as specified on drawing number 17.

connected directly to ground. The center disc is isolated from ground by a machinable glass insulator (1103) and is connected to an external ammeter. A Chromel-Alumel thermocouple mounted in the back of the guard electrode is used for measurement of the flue gas temperature.

Electrical contact to the exposed surface of collected dust layer can be made by lowering a disc electrode (1206) onto the collected dust. The thickness of the layer is determined by comparing the readings of a dial indicator (1003) connected to the movable electrode. The sliding electrode push rod is equipped with a spring to assure that a constant compression force will be applied to the dust layer for each test.

The collected dust layer must be removed by removing the probe from the flue and manually cleaning the electrodes.

## F.3 GENERAL MAINTENANCE

General maintenance of the probe requires that it be periodically disassembled and cleaned. Instructions for maintenance of the electrical equipment are given in the manuals supplied by the manufacturers.

Before disassembling the probe, consult the drawings. In order to clean the probe, first clean and remove the shield (1601) from the point-plane corona discharge assembly. Remove the high voltage and sliding electrode assembly (12B and 13D) from the probe by removing the bolts (1004) on the upper flange and the three screws (1002) holding the high voltage teflon junction block to the middle bulkhead (1404) (plate from which the corona point protrudes). Slide this assembly out of the probe casing and clean.

The high voltage teflon junction block (12-B) consists of two concentric cylinders (1204 and 1205). It can be disassembled by removing the screws (1216) in the side of the junction block. Separating the cylinders exposes the high voltage connection and the sliding electrode contact. This area should be cleaned of any accumulated dust. The graphite contacts (1211) should be checked for good electrical contact and for free motion of the movable electrode (1206).

At the upper end of the high voltage and sliding electrode assembly is the dial indicator assembly (13-D), spring assembly, control mechanisms for lowering the sliding electrode, Swagelok quick connect connector (1324), and the high voltage connector (1312). The dial indicator mechanism has a tendency to corrode and should be very lightly oiled. The vertical location of the dial indicator can be adjusted by losing the locking screw (1308) and sliding the indicator up or down. When the probe is assembled, the dial indicator should be adjusted to read 5.00 when the sliding electrode is in contact with the collecting electrode. The spring assembly should be inspected to insure that the spring operates freely. If it doesn't, dust has probably accumulated in this assembly and it must be disassembled and cleaned. The electrode lowering control (1307) should be free to turn and move up and down when the acme screw is not engaged. The high voltage connector which is fabricated from teflon tubing, Swagelok connectors, and a banana plug, should be cleaned and electrical continuity to the sliding electrode checked.

The collecting electrode electrical connections (11A) are accessible by removing the flange (1102) from the bottom of the probe casing (15A). The machinable glass insulator (1103) which isolates the center disc electrode from ground should be cleaned. The resistance to ground from the center electrode should be greater than $10^{11}$ ohms.

After reassembly, the probe electrode alignment must be inspected. The probe is designed to be self-aligning. In the lowered disc position, the sliding electrode should be parallel and in contact with the center disc electrode. The electrode alignment can be altered slightly by adjusting the three screws (1003) that attach the teflon junction block (1204) to the inner bulkhead.

## F.4 OPERATION OF THE PROBE

### F.4.1 Pre-Field Trip Preparation

Before using the probe in the field, general maintenance should be performed to insure that the probe will operate properly. It is possible to bench-test the probe using the $10^9\ \Omega$ resistor built into the spark protector box to simulate a collected dust layer.

Set up the probe system as described in the next section. Lower the sliding electrode so that it makes contact with the collecting electrode and switch the control on the spark protector to the $10^9\ \Omega$ position. Set the power supply for an output of 100 volts (V) and read the current (I) to the multimeter. Calculate the resistance (R) of the resistcr in the protective box by Ohm's Law:

$$R = \frac{V}{I}$$

This value should be $1.00 \times 10^7\ \Omega \pm 2\%$. Electrical connectors and instrument calibration should be checked if the above value is not obtained.

A pre-field inspection check-list is given in Table F1.

### F.4.2 Operating Instructions

At the test site, the equipment should be carefully unpacked and inspected. The electrical instrumentation package is not sealed to keep out moisture and must be located out of the weather but within 3 m (10 ft) of the sampling port. Connect the probe to ground. This is necessary to insure proper operation of the probe, and for operator safety. Before inserting the probe in the sampling port, lower the sliding electrode until it makes contact with the collecting electrode. If the metal shield (1601) for the corona discharge cell has been removed; replace it at this time. (Between runs it is necessary to remove this shield to clean dust from the cell.) Adapters should be fabricated for 6" and 4" pipe nipples. For some sampling ports special adapting flanges must be made, or a temporary arrangement, such as rags or other suitable sealing material, will have to be used. However, for strongly negative or positive pressure flues, airtight flange connectors should be used.

Insert the probe into the flue with the holes in the cell cover perpendicular to the gas flow and allow the cell to reach flue temperature. Approximately thirty minutes will be required to reach this temperature.

## TABLE F1

## RESISTIVITY PROBE

## PRE-FIELD TRIP INSPECTION CHECK LIST

| YES | NO | | |
|---|---|---|---|
| ____ | ____ | 1. | Probe - Breakdown inspection and calibration - wiring - HV cable, etc. |
| ____ | ____ | 2. | Power supply - Inspect operation - general condition, wiring, calibration, etc. |
| ____ | ____ | 3. | Multimeter - Inspect operation - general condition, wiring, calibration, etc. |
| ____ | ____ | 4. | Tool box - Correct tools for in-field breakdown repair-inventory spare parts. |
| ____ | ____ | 5. | Power cords - Continuity of extension cord and box. |
| ____ | ____ | 6. | Tent - Covering for instruments. |
| ____ | ____ | 7. | Field cleaning kit - Rags, brushes, and dusters for on-site cleaning. |
| ____ | ____ | 8. | Sample containers and data sheets - Supply of bottles or plastic bags to collect ash samples-supply of data sheets. |
| ____ | ____ | 9. | Shipping boxes - Serviceable and in condition to receive rough and abusive handling. Insure that instruments are sufficiently padded. |
| ____ | ____ | 10. | Confirmation - Unusual conditions at test site accounted for: flue gas temperature, gas velocity, flue pressure, sampling port sizes, hot/cold weather conditions, etc. |

Comments: ______________________________

______________________________

______________________________

______________________________

______________________________

A layout of the operating controls on the instrument package is shown in Figure F1. Plug the AC line from the instrumentation package into a 117-120 VAC line. Using their individual power switches, turn on the multimeter and the high voltage supply.

A suitable temperature indicator for a Chromel-Alumel thermocouple should be connected to the thermocouple output on the side of the probe (1506).

After enough time has passed to allow the cell to equilibrate at the flue gas temperature, a test may be started. Twist the operating knob to insure the sliding electrode is lowered down in place. With the probe cover holes oriented perpendicular to the gas stream, unscrew the sliding electrode control and raise it to the up position. Lock in place. Now run a "clean-plate" V-I curve by placing the multimeter on the 100 nA scale and setting the VM switch to the high position. Check that the slide switch on the spark protector is in the normal position. Press the HV ON push button; the red HV light should be on to show the high voltage supply is activated. The use of the high voltage supply is described in the manufacturer's manual. Adjust the OUTPUT control through its full range using the kV meter as a guide and make a current reading every 1000 volts until a spark level or the maximum output voltage is reached. Keep the multimeter within its range during these measurements to prevent excessive overranging. Record these readings on a data sheet and mark it "clean plate". Adjust the HV output control for a current of 2 μA and rotate the probe so the cover plate holes face into the gas stream.

A dust layer is now precipitated on the collecting electrode. The proper operating current density required for the type of dust being collected has to be experimentally determined. Thus, the first test may not be useful for obtaining

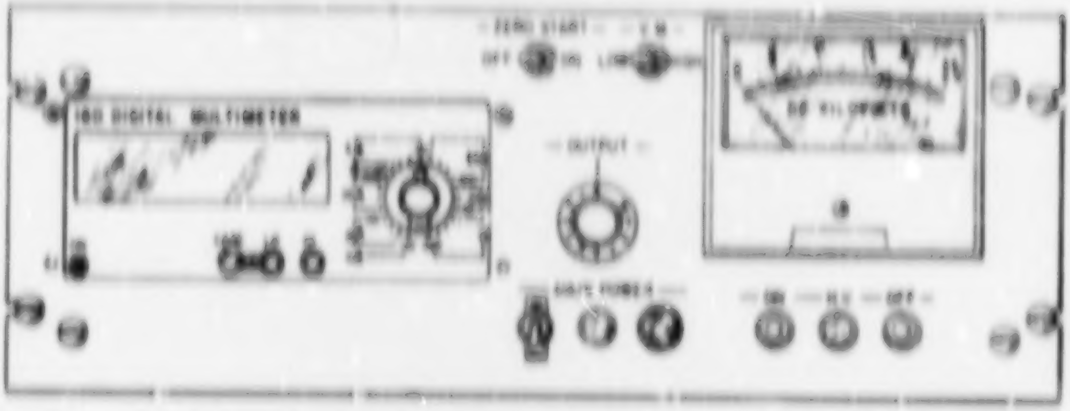
ZERO START
OFF
ON
DIGITAL MULTIMETER
OUTPUT
DC KILOVOLTS
ON
OFF

*Figure F3. Instrument package.*

data. The current density normally used should fall somewhere in the range between 0.2 and 2.0 $\mu A/cm^2$. If a high resistivity dust is encountered, reduced current densities may be required for proper operation. Use of the V-I curves will be explained later to indicate how the proper current for precipitation may be found if the originally selected value proves to be insufficient. A current of 2 $\mu A$, giving a current density of 0.4 $\mu A/cm^2$, is a good place to start the initial test. The voltage necessary to obtain this current will be approximately of 15,000 V. Depending on the resistivity of the dust being collected, the grain loading, and the current density selected, about thirty minutes to one hour may be required to precipitate a sample of thickness between 0.5 and 1.5 mm.

As the dust layer is being deposited, the current will begin to decrease. This current drop may be used to estimate the collection time. When current drops significantly, or if an hour has passed, the test should be stopped. If too small a sample was collected on a short time run, run longer the next time, no matter how much the current happens to decrease. After a sufficient sampling time has elapsed, turn the probe so that the holes in the cover plate are perpendicular to the gas stream. Now run a "dirty-plate" V-I curve using the same procedure as that for the "clean-plate" V-I.

After completing the "dirty-plate" V-I, turn the high voltage off by turning the HV output control to zero and pressing the HV OFF pushbutton. Switch the slide switch on the spark protector box to the $10^9 \Omega$ position. This inserts a $10^9 \Omega$ resistor in the circuit and protects the multimeter from an overload current when lowering the sliding electrode with the voltage on. Set the multimeter on the 100 nA range. Turn the voltage supply on and adjust for a 100 V output.

Unlock and lower the sliding electrode carefully and slowly until the acme screw is engaged. Turn the control to lower the electrode until the multimeter indicates that electrical contact with the dust layers has been made. Turn the control knob one-half turn and stop. If the dust resistivity is less than about $10^7$ Ω-cm, the multimeter should read approximately 100 nA. For high resistivity dust, smaller currents will be obtained, the exact current depending on the thickness of the dust layer and the resistivity. Now set the multimeter on the 1000 μA scale and switch the slide switch on the spark protector back to the normal position. If the power supply does not indicate an overload (4 mA), "spark data" can be taken.* Increase the voltage across the dust layer in 100 V steps. Read and record the corresponding currents until a spark occurs across the dust layer. This will be indicated by the voltmeter needle jumping and an erratic reading on the multimeter.

Before starting another run the dust layer must be removed from the electrodes by mechanically removing the dust.

Remove the probe from the flue. Remove the metal cover from the discharge cell and clean the cell thoroughly. If the sample is to be saved, place a sheet of paper under the disc to collect the sample when the operating rod is pulled back to its up and locked position.

After cleaning, replace the metal cover on the probe. Return the probe to the sampling port. While the probe is returning to the flue temperature, perform calculations for the test just completed.

---

*Overloads frequently occur with high carbon content samples. The carbon particles or similar type conductors provide a conducting path between the disc, allowing the full output current of the power supply to flow. If a short is encountered, it is impossible to obtain data for determining the resistivity of the layer between the parallel discs.

### F.4.3 Operating Outline

1. Prepare sampling port
2. Clean and align cell
3. Lower disc and lock
4. Insert into flue, with inlet holes 90 degrees to flow, and bolt to flange
5. Allow cell to reach flue gemperature
6. Zero dial indicator
7. Raise operating rod
8. Run "clean-plate" V-I, slide switch normal position
9. Turn inlet holes into flow
10. Apply necessary voltage to supply precipitating current
11. After desired length of time turn probe so inlet holes are again 90 degrees to flow. (Leave high voltage applied so dust layer will not be shaken off in the turning process.)
12. Run "dirty-plate" V-I
13. Lower disc, slide switch 10<sup>4</sup> position, voltage 100
14. Record thickness of dust layer
15. Apply "spark-data" voltage 100 V steps until sparkover occurs, slide switch normal position
16. Remove probe or remove collected dust by pressurizing probe
17. Observe layer and save if needed
18. Clean probe and check alignment
19. Insert back into flue
20. Make calculations

### F.4.4 Calculations

A sample data sheet for a typical run is given in Figure F4. All the information necessary for making the resistivity calculations is given on this data sheet. The "clean" and "dirty" plate V-I information should be graphed. The curves for data on this sheet are shown plotted in Figure F5.

The formula for calculating the resistivity is:

$$\rho = \frac{RA}{\ell} \qquad \text{or}$$

$$\rho = \frac{\frac{\Delta V}{I} \times 5.00\ \text{cm}^2}{\ell\ (\text{cm})}$$

where $\rho$ = resistivity (ohm-cm),

$R$ = resistance V/I (ohms),

$\Delta V$ = voltage across the dust layer (volts),

$I$ = measured current (amps),

$A$ = area of disc (5.00 $\text{cm}^2$), and

$\ell$ = thickness of dust layer (cm).

The term $A/\ell$ is called the cell factor. This factor will remain constant for the V-I or spark calculation for each individual run. For different dust layers it is apparent that the cell factor will change.

### F.4.5 Example

The following data were obtained from V-I curves in Figure F5.

## SRI POINT PLANE PROBE DATA

Location : Power Plant Layer Thickness : 1.0 mm
Time : 0915 Date : 14 May 1973 Test No. : A-6 Temp. : 314 F
Conditions : Normal, full load 50 MW
Unit 1, Port 3

| V-I DATA | | |
|---|---|---|
| KV | CLEAN | DIRTY |
| 1 | | |
| 2 | | |
| 3 | | |
| 4 | | |
| 5 | 1.0 NA | 1.0 NA |
| 6 | 0.25 µA | 0.1 µA |
| 7 | 0.65 µA | 0.3 µA |
| 8 | 1.15 µA | 0.5 µA |
| 9 | 1.8 µA | 1.1 µA |
| 10 | 2.6 µA | 1.65 µA |
| 11 | 3.2 µA | 2.10 µA |
| 12 | 4.3 µA | 2.8 µA |
| 13 | 5.1 µA | 3.7 µA |
| 14 | 6.2 µA | 4.2 µA |
| 15 | 7.1 µA | 4.8 µA |
| 16 | 8.2 µA | 5.6 µA |
| 17 | 9.5 µA | 6.25 µA |
| 18 | 11.1 µA | SPARK |
| 19 | 12.6 µA | |
| 20 | SPARK | |

| SPARK DATA | | |
|---|---|---|
| V | I | E |
| 50 | 2.5 NA | 500 |
| 100 | 5.0 NA | 1000 |
| 150 | 7.5 NA | 1500 |
| 200 | 10.0 NA | 2000 |
| 250 | 13.5 NA | 2500 |
| 300 | 17.4 NA | 3000 |
| 350 | 23.6 NA | 3500 |
| 400 | 29.0 NA | 4000 |
| 450 | 39.5 NA | 4500 |
| 500 | 55.5 NA | 5000 |
| 550 | 70.5 NA | 5500 |
| 600 | 98.7 NA | 6000 |
| 650 | 0.14 µA | 6500 |
| 700 | 0.17 µA | 7000 |
| 750 | 0.23 µA | 7500 |
| 800 | 0.36 µA | 8000 |
| 850 | 0.46 µA | 8500 |
| 900 | 0.61 µA | 9000 |
| 950 | 0.75 µA | 9500 |
| 1000 | 1.0 µA | 10,000 |
| 1100 | SPARK | |
| 1200 | | |
| 1300 | | |
| 1400 | | |
| 1500 | | |
| 1600 | | |
| 1700 | | |
| 1800 | | |
| 1900 | | |
| 2000 | | |
| 2100 | | |
| 2200 | | |
| 2300 | | |
| 2400 | | |
| 2500 | | |
| 2600 | | |
| 2700 | | |
| 2800 | | |
| 2900 | | |
| 3000 | | |

3630-075

*Figure F4. Sample data sheet.*

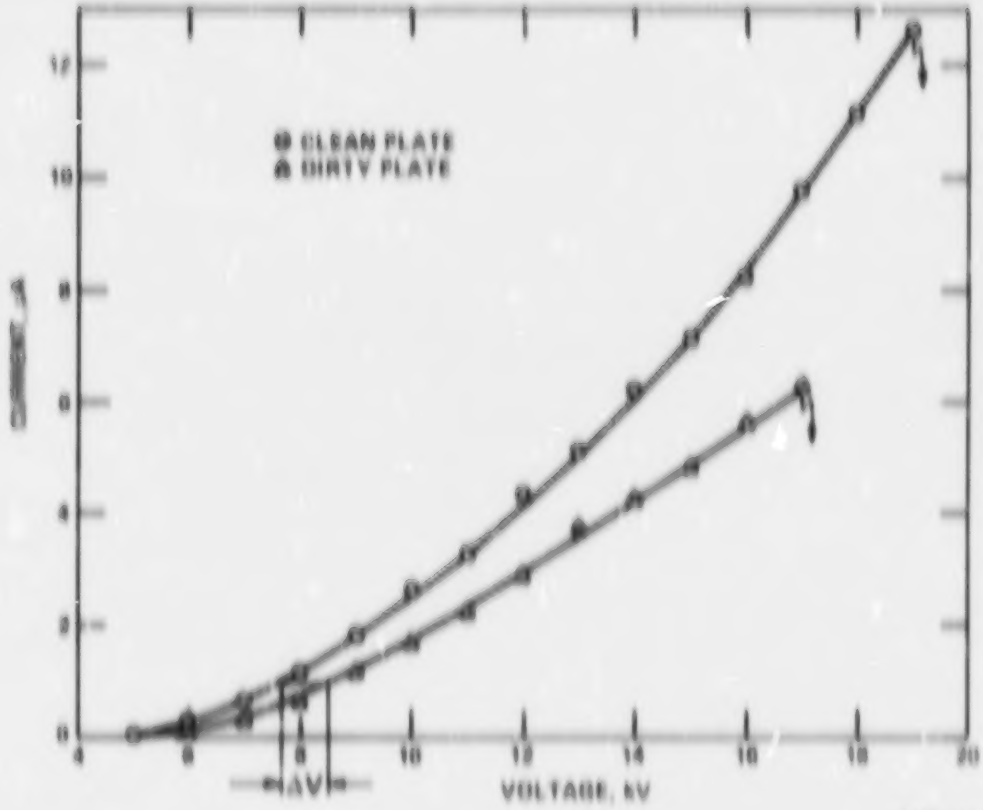

*Figure F8. V-I information.*

$$V = \Delta V = 850V$$
$$I = 1.0x10^{-5}\ A$$
$$\ell = 1.0\ mm = 1.0x10^{-1}\ cm$$

The value V is the voltage drop across the dust layer as interpolated from the V-I curves at a current value of $1.0x10^{-5}$ A. Certain considerations must be taken into account when obtaining this voltage drop. The first is to look at the shape of the V-I curve. There are three basic shapes that may be encountered. The diagrams in Figures F.6. will illustrate two of these shapes.

In Figure F (Top), the point x shows the voltage at which electrical breakdown occurs in the dust layer. This indicates the onset of back corona, a characteristic of a high resistivity dust. It would be incorrect in this case to use any of the current and voltage relationships above the point x for calculating resistivity values.

This V-I curve may be used also to determine the operating point for the next run. If the point x is located at a lower current value than the one selected for collecting the sample then there is a good chance that the sample was collected in a back corona situation. If this is the case then the current for the next run should be reduced to the value of current that corresponds to the point x. More efficient collection should be observed at this setting.

In Figure F6 (Bottom), the "dirty-plate" curve is on the left side of the "clean-plate" curve. This is a characteristic of either a very high or a low-resistivity dust. Since the ΔV taken from the curve will have a negative value, it will not be possible to use the V-I procedure for resistivity calculations in this case. Figure F5 is the third shape and it shows a standard curve.

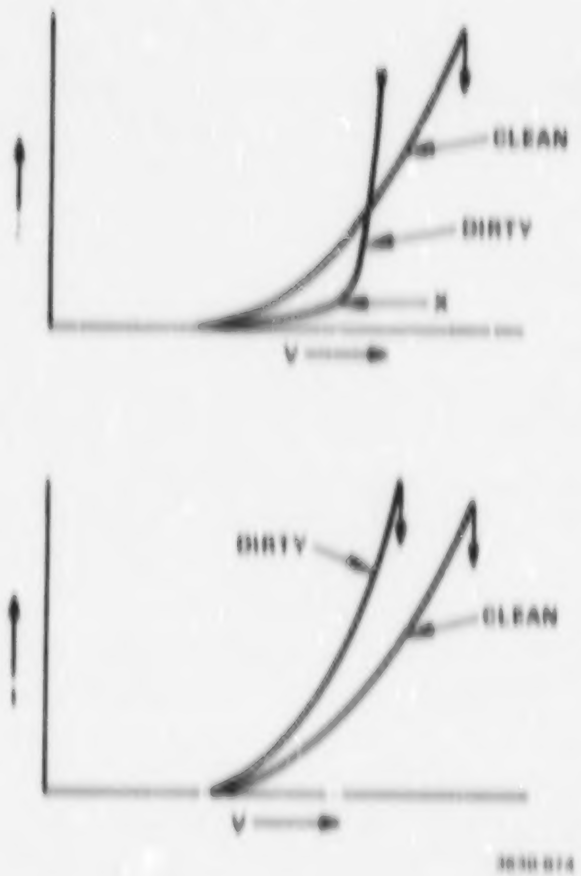

*Figure F6. Two types of clean and dirty plate V-I curves.*

The cell factor is the first calculation to be made. For the sample case, the cell factor is 50 cm and it comes from the term $A/\ell$, where $A$ is 5 cm$^2$ and $\ell$ is 0.1 cm. The next step is to find the resistance $R$ of the dust layer. For this run, $\Delta V$ is equal to 850 V, this was taken from the V-I graph at a current of $1.0 \times 10^{-8}$ amps. From the relation, $R = \Delta V/I$, the resistance is found to be $0.85 \times 10^{11}$ ohm. By multiplying the cell factor by the resistance, a resistivity of $4.2 \times 10^{12}$ ohm-cm is obtained. This complete calculation is:

$$\rho = A/\ell \times V/I$$

or

$$\rho = \frac{5\ \text{cm}^2}{0.1\ \text{cm}} \times \frac{0.85 \times 10^{3}\ \text{V}}{1.0 \times 10^{-8}\ \text{A}}$$

$$\rho = 4.2 \times 10^{12}\ \text{ohm-cm}$$

After obtaining a value for the dust resistivity from the V-I data, a check of this value may be obtained from the spark data information. The proper values to take from the spark data information are the last voltage and current reading before spark. In this case the layer broke down at 1100 volts and the last reading before breakdown was at 1000 volts with a current of $1.0 \times 10^{-8}$ amps. Using the following formula the resistivity data may be obtained:

$$\rho = A/\ell \times V/I$$

$$\rho = \frac{5.0\ \text{cm}^2}{0.1\ \text{cm}} \times \frac{1.0 \times 10^{3}\ \text{V}}{1.0 \times 10^{-8}\ \text{A}}$$

$$\rho = 5.0 \times 10^{12}\ \text{ohm-cm}$$

The column labeled "E" on the spark data sheet is for the calculated electric field for the voltage applied and the thickness of the layer. In this example, breakdown of the layer occurred at an electric field of 10,000 volts/cm. When a series of measurements are made, the resistivities should be calculated not only at sparkover for each run, but also at a fixed value of the electric field. This will eliminate the electric field dependence when comparing runs.

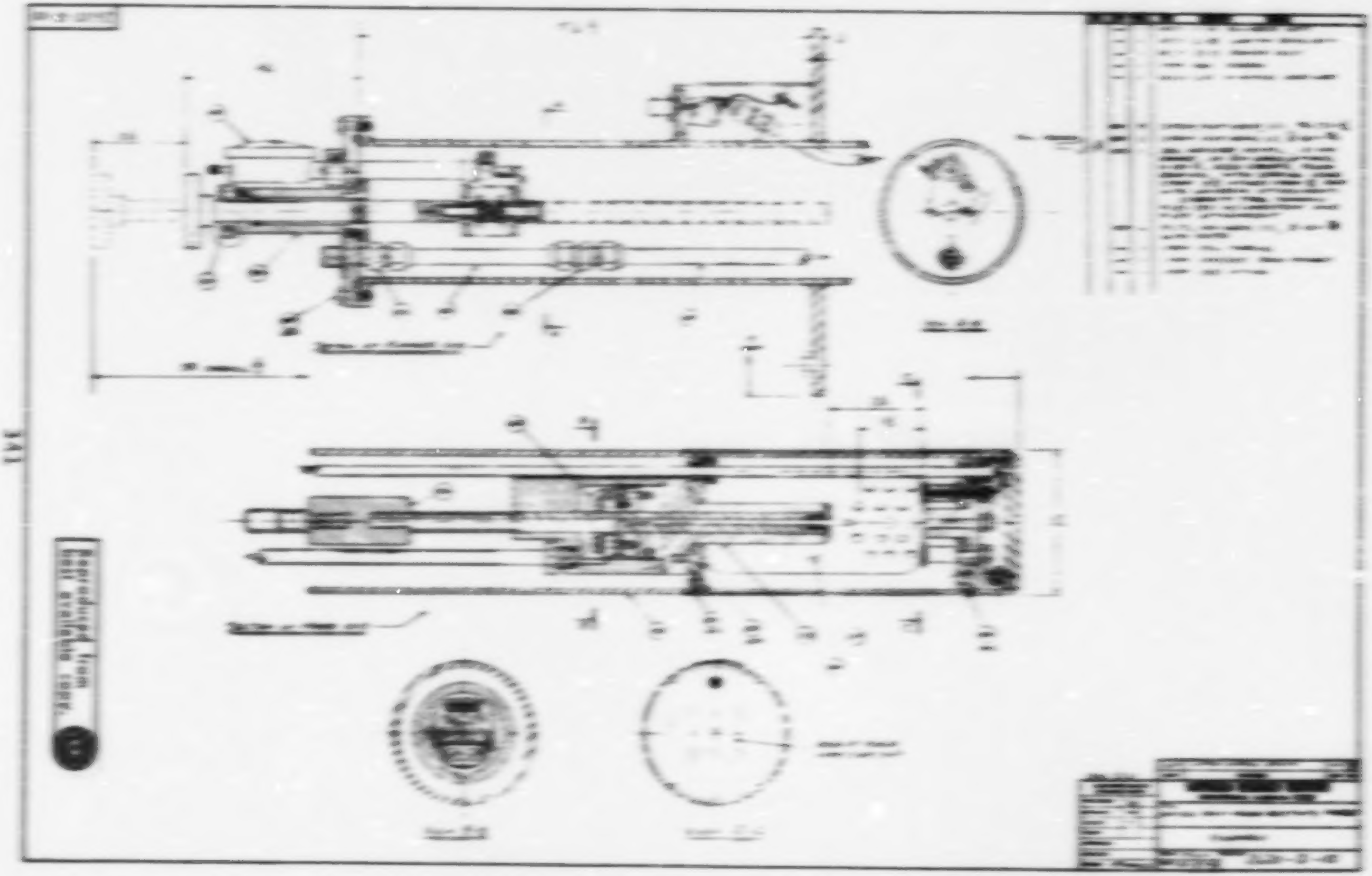

Figure 77. In-situ resistivity probe.

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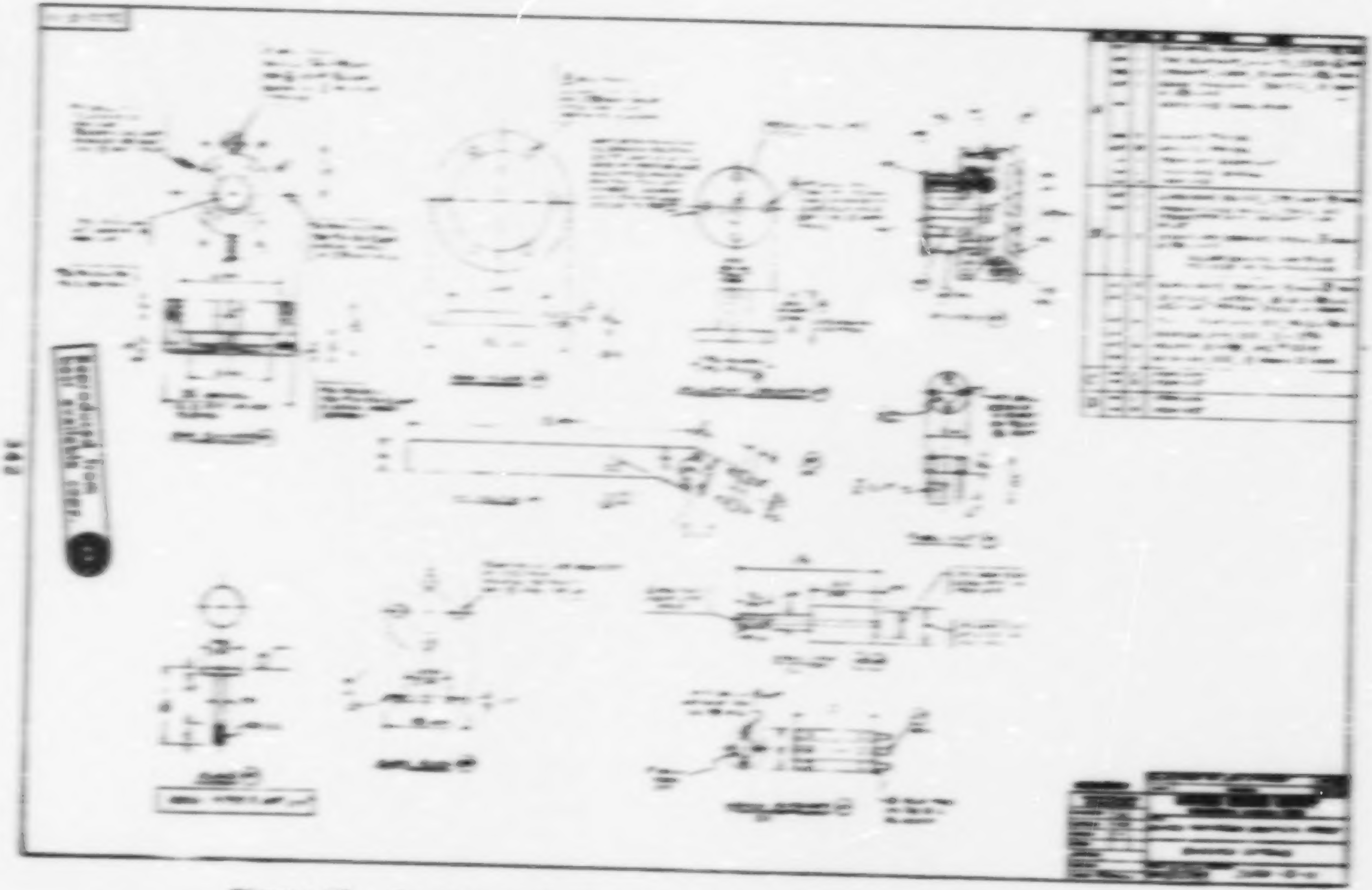

Figure 78. In-situ resistivity probe.

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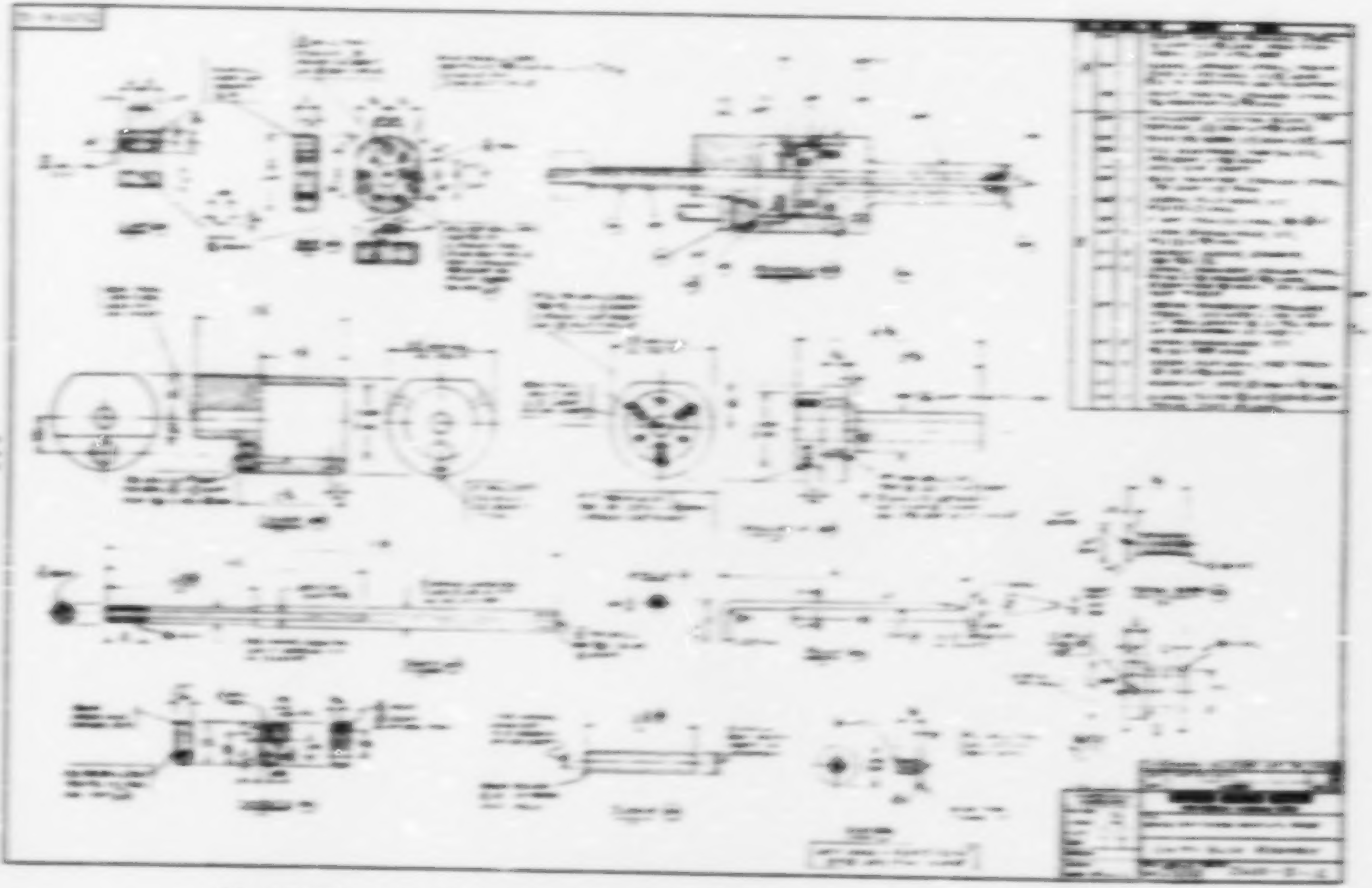

Figure F9. In-situ resistivity probe.

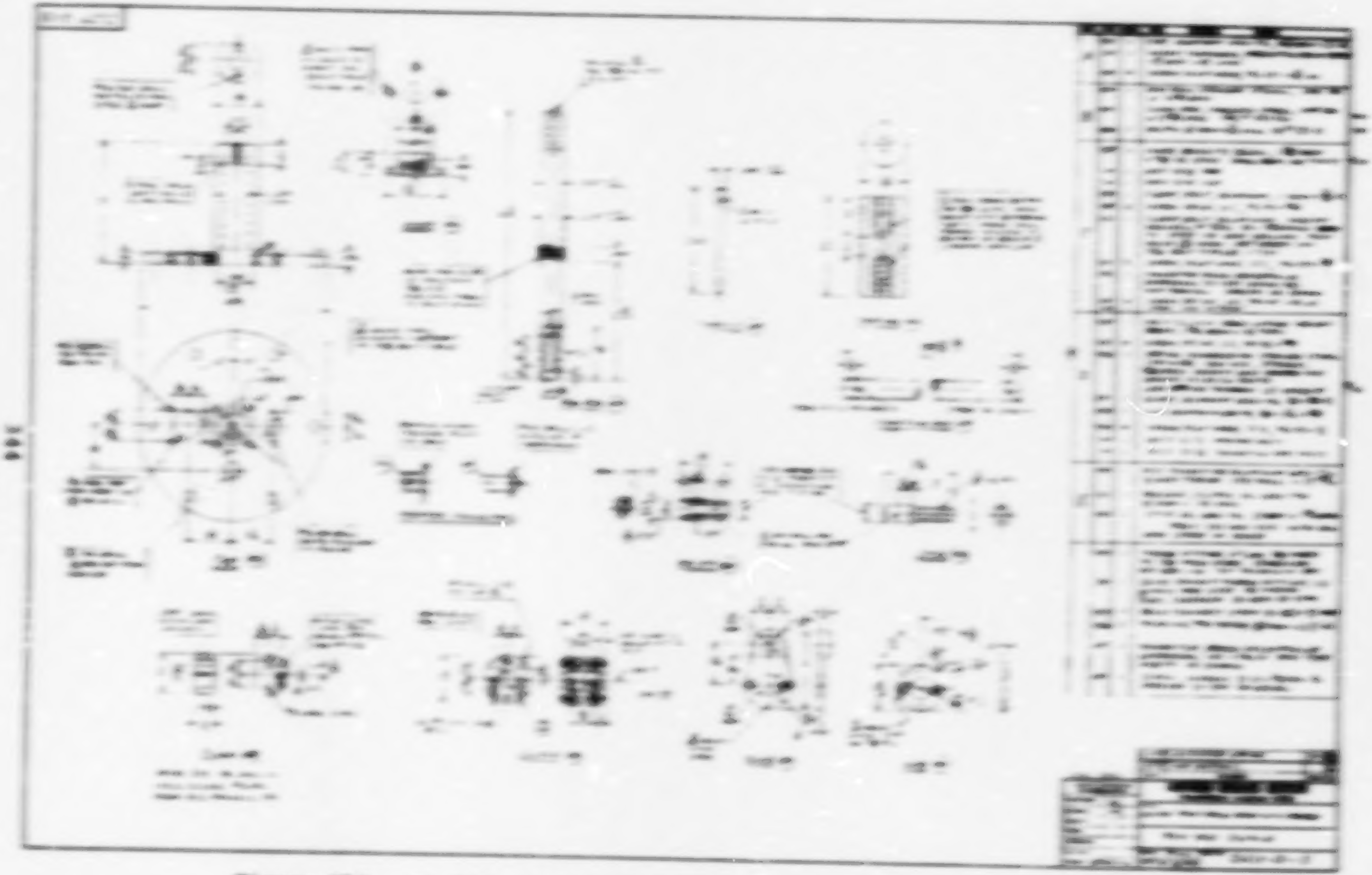

Figure F13. In-situ resistivity probe.

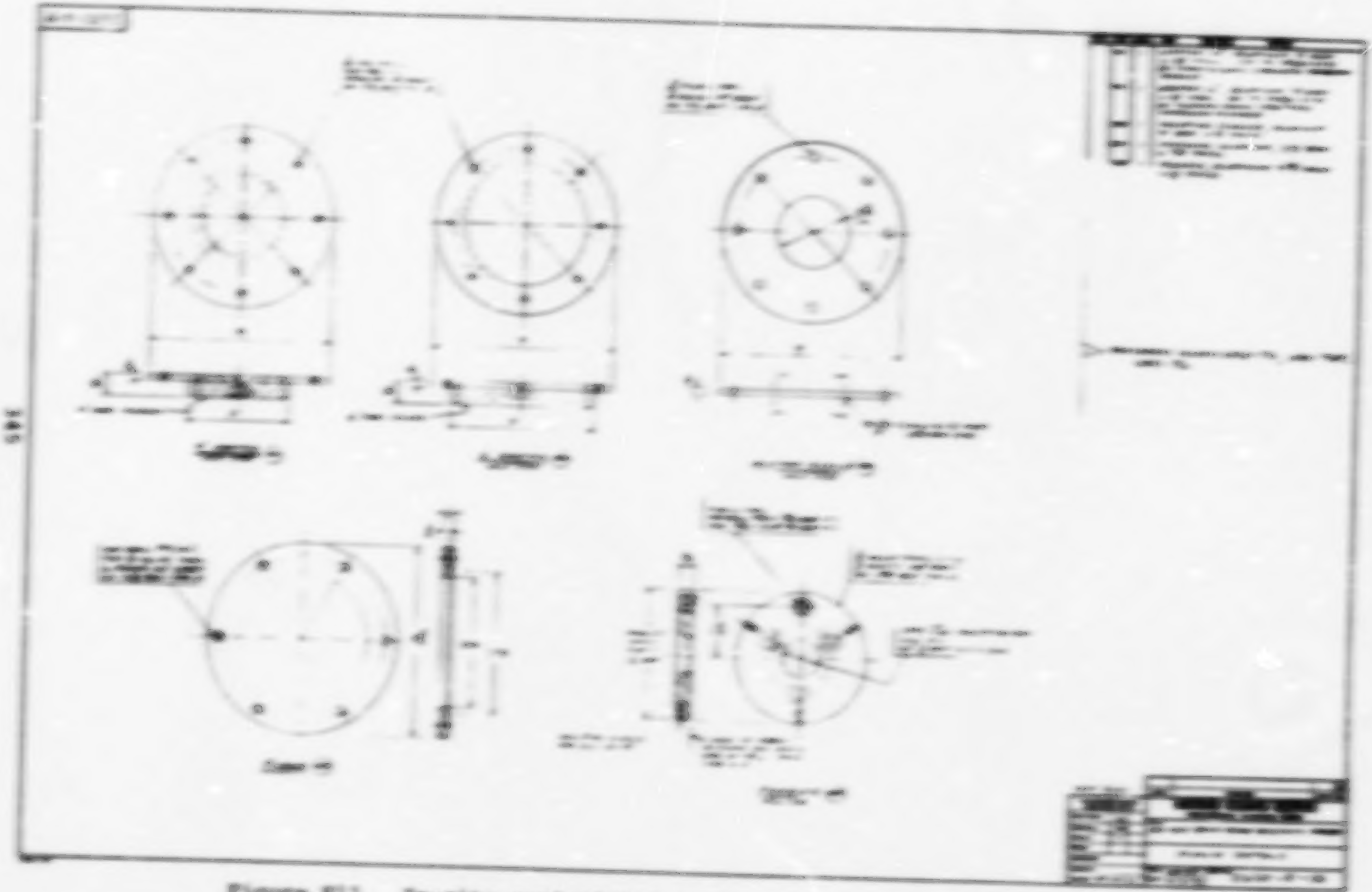

Figure F11. In-situ resistivity probe.

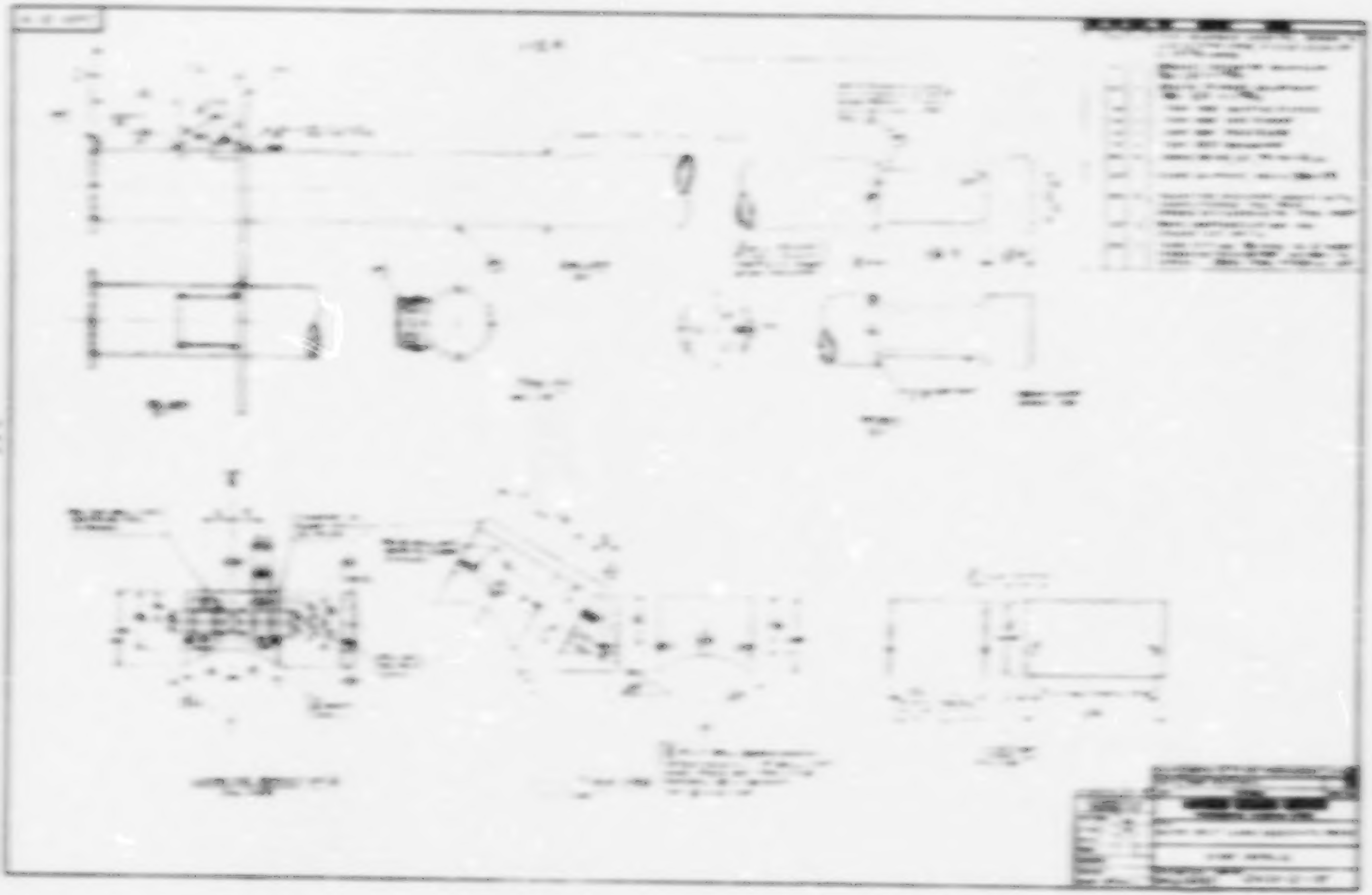

Figure F12. In-situ conductivity probe.

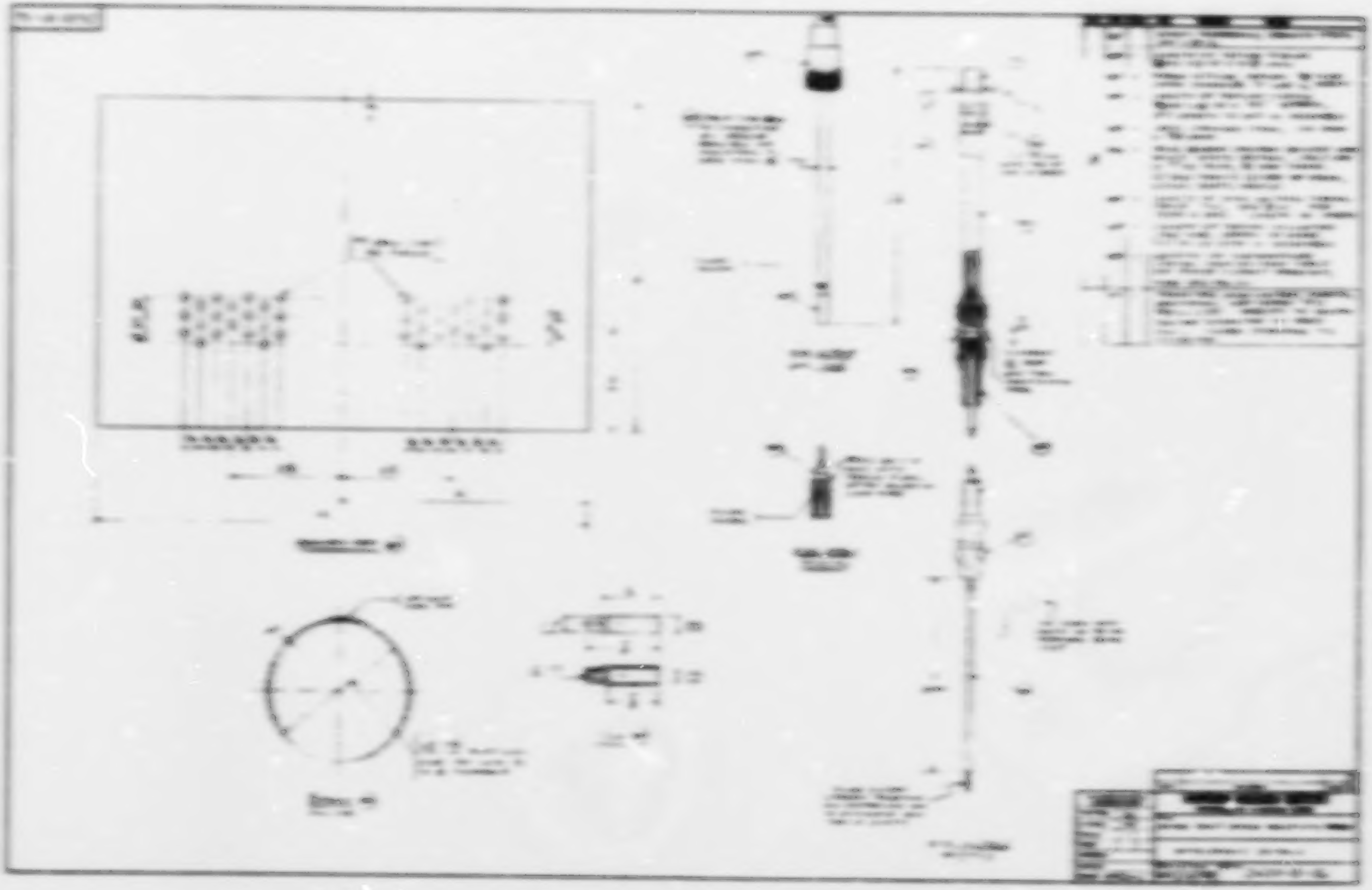

Figure F13. In-situ resistivity probe.

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2. Nichols, Grady B. Techniques for Measuring Fly Ash Resistivity. EPA 650/2-74-079 (August, 1974). U.S. Environmental Protection Agency, Research Triangle, Park, North Carolina.

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# APPENDIX G

## FEDERAL STATIONARY SOURCE PERFORMANCE REFERENCE METHODS

To evaluate the performance standards for new stationary sources, the EPA has promulgated reference methods which specify the manner in which certain tests must be performed. These Reference Methods can be found in the Code of Federal Regulations under Title 40 - Protection of Environment; Chapter I - Environmental Protection Agency; Subchapter C - Air Programs; Part 60 - Standards of Performance for New Stationary Sources; Appendix A - Reference Methods.

In the first section of this appendix these reference methods are summarized. In the later part these reference methods, as presented in the Code of Federal Regulations, are reproduced verbatim. They have been corrected and amended through May 15, 1977.

## METHOD 1

Sample and Velocity Traverses for Stationary Sources:

Procedure for selecting stack sampling site and selecting and locating minimum number of traverse points.

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Regulation Promulgated | 36 | 247 | 12/23/71 | 24882 |
| Regulation Amended | 38 | 99 | 5/23/73 | 13562 |
| Proposed Revised Regulation | 41 | 111 | 6/08/76 | 23061 |

## METHOD 2

Determination of Stack Gas and Volumetric Flowrate:

Procedure for determining stack gas velocity from gas density and velocity head using Type S pitot tube. Volumetric flowrate is calculated from gas velocity and stack cross-sectional area.

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol | No. | Date | Page |
| Regulation Promulgated | 36 | 247 | 12/23/71 | 24886 |
| Proposed Revised Regulation | 41 | 111 | 6/08/76 | 23063 |

## METHOD 3

Gas Analysis for Carbon Dioxide, Excess Air, and Dry Molecular Weight:

$CO_2$, CO, and $O_2$ are determined by absorbing in appropriate reagents (Orsat analysis).

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol., | No. | Date | Page |
| Regulation Promulgated | 36 | 247 | 12/23/71 | 24886 |
| Proposed Revised Regulation | 41 | 111 | 6/08/76 | 23069 |

## METHOD 4

**Determination of Moisture in Stack Gases:**

Stack gas moisture content is determined by condensation and measuring condensed water volumetrically.

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Regulation Promulgated | 36 | 247 | 12/23/71 | 24887 |
| Proposed Revised Regulation | 41 | 111 | 6/08/76 | 23072 |

## METHOD 5

**Determination of Particulate Emissions from Stationary Sources:**

Stack gas is sampled isokinetically, particulate matter filtered and weighed after removal of uncombined water.

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Regulation Promulgated | 36 | 247 | 12/23/71 | 24888 |
| Regulation Amended | 38 | 99 | 5/23/73 | 13563 |
| Proposed Revised Regulation | 41 | 111 | 6/08/76 | 23076 |

## METHOD 6

**Determination of Sulfur Dioxide Emissions from Stationary Sources:**

$SO_2$ is separated from any $SO_3$ and $H_2SO_4$ mist present using aqueous isopropyl alcohol. The $SO_2$ is passed through dilute hydrogen peroxide which is titrated by the barium-thorin method to determine sulfate formation.

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Regulation Promulgated | 36 | 247 | 12/23/71 | 24890 |
| Proposed Revised Regulation | 41 | 111 | 6/08/76 | 23083 |

## METHOD 7

### Determination of Nitrogen Oxide Emissions from Stationary Sources:

A grab sample is drawn into an evacuated flask containing actified hydrogen peroxide which converts NO and $NO_2$ to $HNO_3$. Analysis is made by the colorimetric PDS method.

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Regulation Promulgated | 36 | 247 | 12/23/71 | 24891 |
| Proposed Revised Regulation | 41 | 111 | 6/08/76 | 23085 |

## METHOD 8

### Determination of Sulfuric Acid Mist and Sulfur Dioxide Emissions from Stationary Sources:

$SO_3$ and $H_2SO_4$ mist are absorbed in aqueous isopropyl alcohol and $SO_2$ passes through to be collected in hydrogen peroxide solution. Each solution is titrated by the barium-thorin method to determine sulfate formation.

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Regulation Promulgated | 36 | 247 | 12/23/71 | 24893 |
| Proposed Revised Regulation | 41 | 111 | 6/08/76 | 23087 |

## METHOD 9

### Visual Determination of the Opacity of Emissions from Stationary Sources:

Opacity of Emissions from a stationary source is determined visually by qualified observers.

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Regulation Promulgated | 36 | 247 | 12/23/71 | 24895 |
| Proposed Revised Regulation | 39 | 177 | 9/11/74 | 32857 |
| Revised Regulation Promulgated | 39 | 219 | 11/12/74 | 39874 |

## METHOD 10

### Determination of Carbon Monoxide Emissions from Stationary Sources:

Integrated or continuous gas samples from stacks are analysed for CO content using NDIR analysis.

| | | Federal Register | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Regulation Promulgated | 39 | 47 | 3/08/74 | 9319 |
| Regulation Amended | 39 | 75 | 4/07/74 | 13776 |

## METHOD 11

### Determination of Hydrogen Sulfide Emissions from Stationary Sources:

$H_2S$ is collected in alkaline $Cd(OH)_2$ to form CdS, which is dissolved in HCL and reacted with a known amount of iodine. Amount of iodine consumed is measure of $H_2S$ present in gas.

| | | Federal Register | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Regulation Promulgated | 39 | 47 | 3/08/74 | 9321 |
| Regulation Amended | 39 | 75 | 4/17/74 | 13776 |
| Proposed Revised Regulation | 42 | 99 | 5/23/77 | 26222 |

## METHOD 12

### Determination of Sulfur Dioxide Emissions from Stationary Sources by Continuous Monitors:

No specific continuous monitor required; results must show acceptable relationship to those determined by Method 6 or 8.

(RESERVED)

## METHOD 13A

Determination of Total Fluoride Emissions from Stationary Sources - SPADNS Zirconium Lake Method:

Gaseous and particulate fluorides are withdrawn isokinetically and concentration determined by SPADNS method.

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Regulation Promulgated | 40 | 152 | 8/06/75 | 33157 |
| Regulation Amended | 41 | 230 | 11/29/76 | 52299 |

## METHOD 13B

Determination of Total Fluoride Emissions from Stationary Sources - Specific Ion Electrode Method:

Gaseous and particulate fluorides are withdrawn isokinetically and concentration determined by a specific ion electrode.

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Regulation Promulgated | 40 | 152 | 8/06/75 | 33163 |
| Regulation Amended | 41 | 230 | 11/29/76 | 52299 |

## METHOD 14

Determination of Fluoride Emissions from Pot Room Roof Monitors of Primary Aluminum Plants:

A permanent sampling manifold is constructed to isokinetically withdraw sample gas from the roof monitor. The gas sample is brought to ground level and sampled using Method 13A or 13B for determination of fluoride concentration.

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Regulation Promulgated | 41 | 17 | 1/26/76 | 3829 |

## METHOD 15 (Proposed)

Determination of $H_2S$, COS, and $CS_2$ Emissions from Stationary Sources:

A gas sample is extracted and diluted with clean dry air. An aliquot of the diluted sample is then analysed by gas chromatographic separation (GC) and flame photometric detection (FPD).

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Proposed Regulation | 41 | 193 | 10/04/76 | 43870 |

## METHOD 16 (Proposed)

Semicontinuous Determination of Sulfur Emissions from Stationary Sources:

A gas sample is extracted and diluted with clean dry air. An aliquot of the diluted sample is then analysed for gas sulfur. Two GC/FPD analysers are used for resolution of both high and low molecular weight sulfur compounds.

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Proposed Regulation | 41 | 187 | 9/24/76 | 42017 |

## METHOD 17 (Proposed)

Determination of Particulate Emissions from Stationary Sources (In-Stack Filtration Method):

Particulate matter is withdrawn isokinetically and collected on a filter maintained at stack temperature. Particulate matter mass is determined gravimetrically after removal of uncombined $H_2O$.

| | Federal Register | | | |
|---|---|---|---|---|
| | Vol. | No. | Date | Page |
| Proposed Regulation | 41 | 187 | 9/24/76 | 42020 |

# METHOD 1

## Sample and Velocity Traverses For Stationary Sources

1. Principle and Applicability.

1.1 Principle. A sampling site and the number of traverse points are selected to aid in the extraction of a representative sample.

1.2 Applicability. This method should be applied only when specified by the test procedures for determining compliance with the New Source Performance Standards. Unless otherwise specified, this method is not intended to apply to gas streams other than those emitted directly to the atmosphere without further processing.

2. Procedure.

2.1 Selection of a sampling site and minimum number of traverse points.

2.1.1 Select a sampling site that is at least eight stack or duct diameters downstream and two diameters upstream from any flow disturbance such as a bend, expansion, contraction, or visible flame. For rectangular cross section, determine an equivalent diameter from the following equation:

$$\text{equivalent diameter} = 2\left(\frac{(\text{length})(\text{width})}{\text{length} + \text{width}}\right)$$

equation 1-1

2.1.2 When the above sampling site criteria can be met, the minimum number of traverse points is twelve (12).

2.1.3 Some sampling situations render the above sampling site criteria impractical. When this is the case, choose a convenient sampling location and use Figure 1-1 to determine the minimum number of traverse points. Under no conditions should a sampling point be selected within 1 inch of the stack wall. To obtain the number of traverse points for stacks or ducts with a diameter less than 2 feet, multiply the number of points obtained from Figure 1-1 by 0.67.

2.1.4 To use Figure 1-1 first measure the distance from the chosen sampling location to the nearest upstream and downstream disturbances. Determine the corresponding number of traverse points for each distance from Figure 1-1. Select the higher of the two numbers of traverse points, or a greater value, such that for circular stacks the number is a multiple of 4, and for rectangular stacks the number follows the criteria of section 2.2.2.

2.2 Cross-sectional layout and location of traverse points.

2.2.1 For circular stacks locate the traverse points on at least two diameters according to Figure 1-2 and Table 1-1. The traverse axes shall divide the stack cross section into equal parts.

2.2.2 For rectangular stacks divide the cross section into as many equal rectangular areas as traverse points, such that the ratio of the length to the width of the elemental areas is between one and two. Locate the traverse points at the centroid of each equal area according to Figure 1-3.

3. References.

Determining Dust Concentration in a Gas Stream, ASME Performance Test Code #27, New York, N.Y., 1957.

Devorkin, Howard, et al., Air Pollution Source Testing Manual, Air Pollution Control District, Los Angeles, Calif., November 1963.

Methods for Determination of Velocity, Volume, Dust and Mist Content of Gases, Western Precipitation Division of Joy Manufacturing Co., Los Angeles, Calif., Bulletin WP-50, 1968.

Standard Method for Sampling Stacks for Particulate Matter, In: 1971 Book of ASTM Standards, Part 23, Philadelphia, Pa. 1971, ASTM Designation D-2928-71.

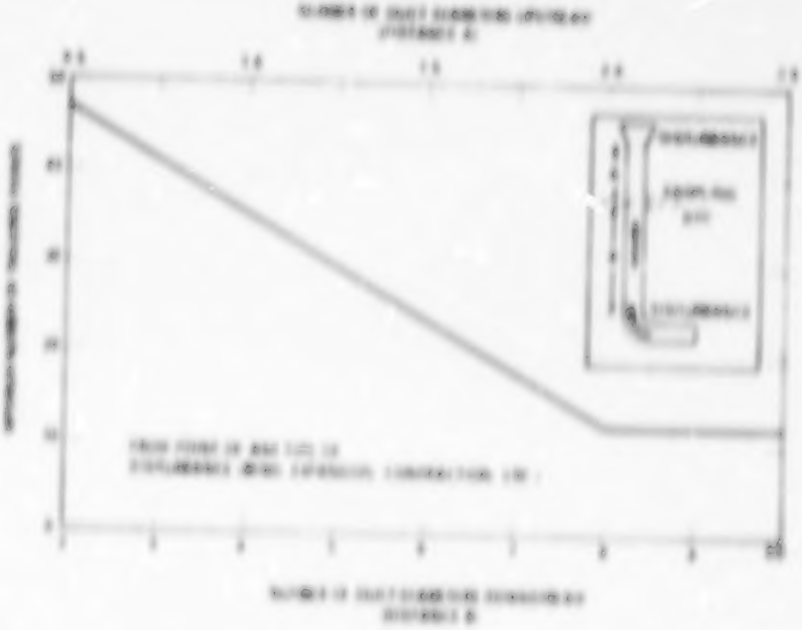

Figure 1-1. Minimum number of traverse points.

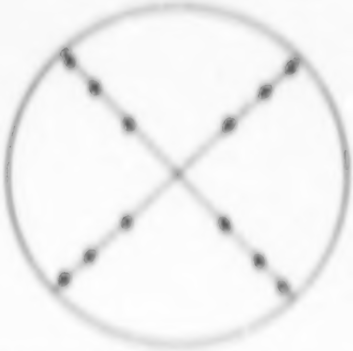

Figure 1-2. Cross section of circular stack divided into 12 equal areas, showing location of traverse points at centroid of each area.

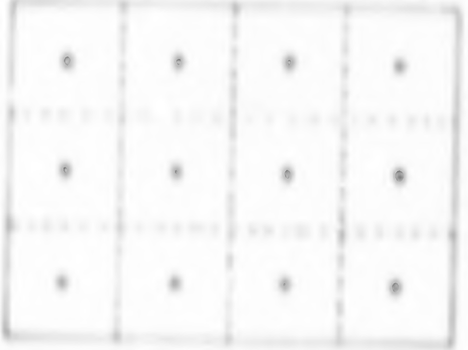

Figure 1-3. Cross section of rectangular stack divided into 12 equal areas, with traverse points at centroid of each area.

Table 1-1. Location of traverse points in circular stacks
(Percent of stack diameter from inside wall to traverse point)

| Traverse point number on a diameter | Number of traverse points on a diameter | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 2 | 4 | 6 | 8 | 10 | 12 | 14 | 16 | 18 | 20 | 22 | 24 |
| 1 | 14.6 | 6.7 | 4.4 | 3.2 | 2.6 | 2.1 | 1.8 | 1.6 | 1.4 | 1.3 | 1.1 | 1.1 |
| 2 | 85.4 | 25.0 | 14.6 | 10.5 | 8.2 | 6.7 | 5.7 | 4.9 | 4.4 | 3.9 | 3.5 | 3.2 |
| 3 | | 75.0 | 29.6 | 19.4 | 14.6 | 11.8 | 9.9 | 8.5 | 7.5 | 6.7 | 6.0 | 5.5 |
| 4 | | 93.3 | 70.4 | 32.3 | 22.6 | 17.7 | 14.6 | 12.5 | 10.9 | 9.7 | 8.7 | 7.9 |
| 5 | | | 85.4 | 67.7 | 34.2 | 25.0 | 20.1 | 16.9 | 14.6 | 12.9 | 11.6 | 10.5 |
| 6 | | | 95.6 | 80.6 | 65.8 | 35.6 | 26.9 | 22.0 | 18.8 | 16.5 | 14.6 | 13.2 |
| 7 | | | | 89.5 | 77.4 | 64.4 | 36.6 | 28.3 | 23.6 | 20.4 | 18.0 | 16.1 |
| 8 | | | | 96.8 | 85.4 | 75.0 | 63.4 | 37.5 | 29.6 | 25.0 | 21.8 | 19.4 |
| 9 | | | | | 91.8 | 82.3 | 73.1 | 62.5 | 38.2 | 30.6 | 26.2 | 23.0 |
| 10 | | | | | 97.4 | 88.2 | 79.9 | 71.7 | 61.8 | 38.8 | 31.5 | 27.2 |
| 11 | | | | | | 93.3 | 85.4 | 78.0 | 70.4 | 61.2 | 39.3 | 32.3 |
| 12 | | | | | | 97.9 | 90.1 | 83.1 | 76.4 | 69.4 | 60.7 | 39.8 |
| 13 | | | | | | | 94.3 | 87.5 | 81.2 | 75.0 | 68.5 | 60.2 |
| 14 | | | | | | | 98.2 | 91.5 | 85.4 | 79.6 | 73.8 | 67.7 |
| 15 | | | | | | | | 95.1 | 89.1 | 83.5 | 78.2 | 72.8 |
| 16 | | | | | | | | 98.4 | 92.5 | 87.1 | 82.0 | 77.0 |
| 17 | | | | | | | | | 95.6 | 90.3 | 85.4 | 80.6 |
| 18 | | | | | | | | | 98.6 | 93.3 | 88.4 | 83.9 |
| 19 | | | | | | | | | | 96.1 | 91.3 | 86.8 |
| 20 | | | | | | | | | | 98.7 | 94.0 | 89.5 |
| 21 | | | | | | | | | | | 96.5 | 92.1 |
| 22 | | | | | | | | | | | 98.9 | 94.5 |
| 23 | | | | | | | | | | | | 96.8 |
| 24 | | | | | | | | | | | | 98.9 |

# METHOD 2

# Determination Of Stack Gas Velocity And Volumetric Flow Rate (Type S Pitot Tube)

[illegible]

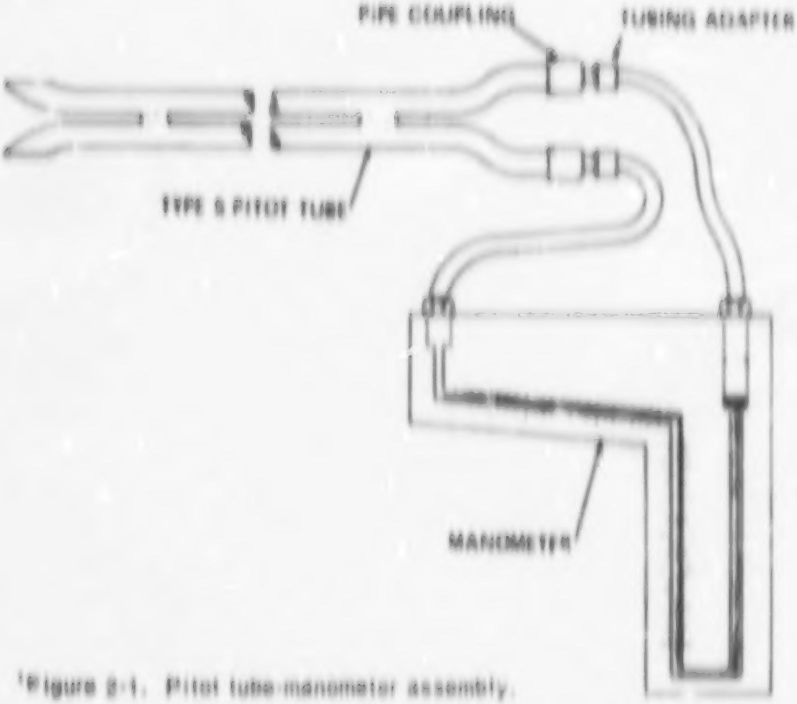
PIPE COUPLING
TUBING ADAPTER
TYPE S PITOT TUBE
MANOMETER

Figure 2-1. Pitot tube-manometer assembly.

PLANT____________________

DATE____________________

RUN NO.____________________

STACK DIAMETER, in.____________________

BAROMETRIC PRESSURE, in. Hg____________________

STATIC PRESSURE IN STACK ($P_g$), in. Hg____________________

OPERATORS____________________

SCHEMATIC OF STACK CROSS SECTION

| Traverse point number | Velocity head, in. $H_2O$ | $\sqrt{\Delta p}$ | Stack Temperature ($t_s$), °F |
|---|---|---|---|
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| | AVERAGE: | | |

Figure 2-2. Velocity traverse data.

# METHOD 3

## Gas Analysis For Carbon Dioxide, Excess Air, And Dry Molecular Weight

*1. Principle and applicability.*

1.1 Principle. An integrated or grab gas sample is extracted from a sampling point and analyzed for its components using an Orsat analyzer.

1.2 Applicability. This method should be applied only when specified by the test procedures for determining compliance with the New Source Performance Standards. The test procedure will indicate whether a grab sample or an integrated sample is to be used.

*2. Apparatus.*

2.1 *Grab sample (Figure 3-1).*

2.1.1 Probe—Stainless steel or Pyrex[1] glass, equipped with a filter to remove particulate matter.

2.1.2 Pump—One-way squeeze bulb, or equivalent, to transport gas sample to analyzer.

2.2 *Integrated sample (Figure 3-2).*

2.2.1 Probe—Stainless steel or Pyrex[1] glass, equipped with a filter to remove particulate matter.

2.2.2 Air-cooled condenser or equivalent—To remove any excess moisture.

2.2.3 Needle valve—To adjust flow rate.

2.2.4 Pump—Leak-free, diaphragm type, or equivalent, to pull gas.

2.2.5 Rate meter—To measure a flow range from 0 to 0.035 cfm.

2.2.6 Flexible bag—Tedlar,[1] or equivalent, with a capacity of 2 to 3 cu. ft. Leak test the bag in the laboratory before using.

2.2.7 Pitot tube—Type S, or equivalent, attached to the probe so that the sampling flow rate can be regulated proportional to the stack gas velocity when velocity is varying with time or a sample traverse is conducted.

2.3 *Analysis.*

2.3.1 Orsat analyzer, or equivalent.

*3. Procedure.*

3.1 *Grab sampling.*

3.1.1 Set up the equipment as shown in Figure 3-1, making sure all connections are leak-free. Place the probe in the stack at a sampling point and purge the sampling line.

3.1.2 Draw sample into the analyzer.

3.2 *Integrated sampling.*

3.2.1 Evacuate the flexible bag. Set up the equipment as shown in Figure 3-2 with the bag disconnected. Place the probe in the stack and purge the sampling line. Connect the bag, making sure that all connections are tight and that there are no leaks.

3.2.2 Sample at a rate proportional to the stack velocity.

3.3 *Analysis.*

3.3.1 Determine the $CO_2$, $O_2$, and CO concentrations as soon as possible. Make as many passes as are necessary to give constant readings. If more than ten passes are necessary, replace the absorbing solution.

3.3.2 For grab sampling, repeat the sampling and analysis until three consecutive samples vary no more than 0.5 percent by volume for each component being analyzed.

3.3.3 For integrated sampling, repeat the analysis of the sample until three consecutive analyses vary no more than 0.2 percent by volume for each component being analyzed.

*4. Calculations.*

4.1 Carbon dioxide. Average the three consecutive runs and report the result to the nearest 0.1% $CO_2$.

4.2 Excess air. Use Equation 3-1 to calculate excess air, and average the runs. Report the result to the nearest 0.1% excess air.

$$\%EA = \frac{(\%O_2) - 0.5(\%CO)}{0.264(\%N_2) - (\%O_2) + 0.5(\%CO)} \times 100$$

equation 3-1

where:

%EA = Percent excess air.

$\%O_2$ = Percent oxygen by volume, dry basis.

$\%N_2$ = Percent nitrogen by volume, dry basis.

%CO = Percent carbon monoxide by volume, dry basis.

0.264 = Ratio of oxygen to nitrogen in air by volume.

4.3 Dry molecular weight. Use Equation 3-2 to calculate dry molecular weight and average the runs. Report the result to the nearest tenth.

$$M_d = 0.44(\%CO_2) + 0.32(\%O_2) + 0.28(\%N_2 + \%CO)$$

equation 3-2

where:

$M_d$ = Dry molecular weight, lb./lb-mole.

$\%CO_2$ = Percent carbon dioxide by volume, dry basis.

$\%O_2$ = Percent oxygen by volume, dry basis.

$\%N_2$ = Percent nitrogen by volume, dry basis.

0.44 = Molecular weight of carbon dioxide divided by 100.

0.32 = Molecular weight of oxygen divided by 100.

0.28 = Molecular weight of nitrogen and CO divided by 100.

*5. References.*

Altshuller, A. P., et al., Storage of Gases and Vapors in Plastic Bags, Int. J. Air & Water Pollution, 6:75-81, 1963.

Conner, William D., and J. S. Nader, Air Sampling with Plastic Bags, Journal of the American Industrial Hygiene Association, 25:291-297, May-June 1964.

Devorkin, Howard, et al., Air Pollution Source Testing Manual, Air Pollution Control District, Los Angeles, Calif., November 1963.

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[1] Trade name.

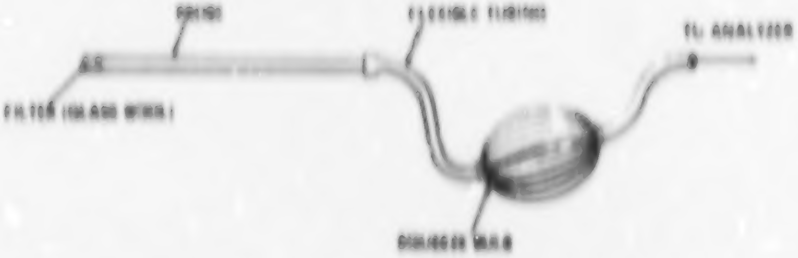
PROBE
FLEXIBLE TUBING
TO ANALYZER
FILTER (GLASS WOOL)
SQUEEZE BULB

Figure 3-1. Grab-sampling train.

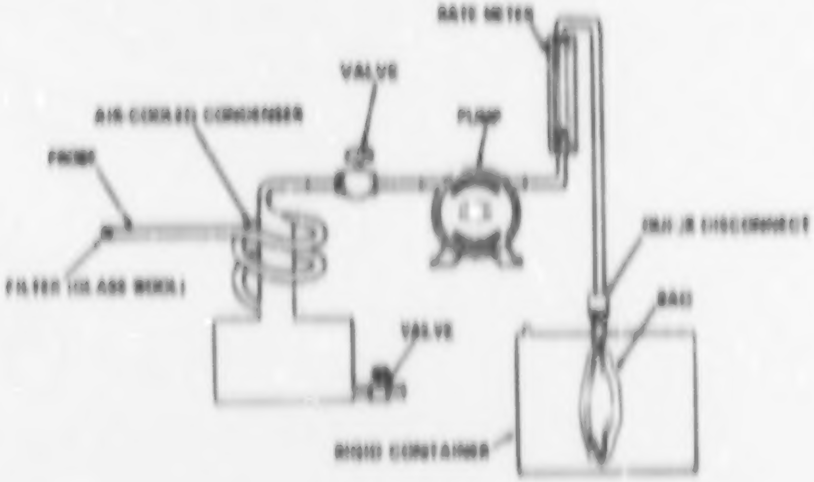
RATE METER
VALVE
AIR-COOLED CONDENSER
PUMP
PROBE
QUICK DISCONNECT
FILTER (GLASS WOOL)
BAG
VALVE
RIGID CONTAINER

Figure 3-2. Integrated gas - sampling train.

# METHOD 4

## Determination Of Moisture In Stack Gases

1. Principle and applicability.

1.1 Principle. Moisture is removed from the gas stream, condensed, and determined volumetrically.

1.2 Applicability. This method is applicable for the determination of moisture in stack gas only when specified by test procedures for determining compliance with New Source Performance Standards. This method does not apply when liquid droplets are present in the gas stream[1] and the moisture is subsequently used in the determination of stack gas molecular weight.

Other methods such as drying tubes, wet bulb-dry bulb techniques, and volumetric condensation techniques may be used.

2. Apparatus.

2.1 Probe—Stainless steel or Pyrex[2] glass sufficiently heated to prevent condensation and equipped with a filter to remove particulate matter.

2.2 Impingers—Two midget impingers, each with 30 ml. capacity, or equivalent.

2.3 Ice bath container—To condense moisture in impingers.

2.4 Silica gel tube (optional)—To protect pump and dry gas meter.

2.5 Needle valve—To regulate gas flow rate.

2.6 Pump—Leak-free, diaphragm type, or equivalent, to pull gas through train.

2.7 Dry gas meter—To measure to within 1% of the total sample volume.

2.8 Rotameter—To measure a flow range from 0 to 0.1 c.f.m.

2.9 Graduated cylinder—25 ml.

2.10 Barometer—Sufficient to read to within 0.1 inch Hg.

2.11 Pitot tube—Type S, or equivalent, attached to probe so that the sampling flow rate can be regulated proportional to the stack gas velocity when velocity is varying with time or a sample traverse is conducted.

3. Procedure.

3.1 Place exactly 5 ml. distilled water in each impinger. Assemble the apparatus without the probe as shown in Figure 4-1. Leak check by plugging the inlet to the first impinger and drawing a vacuum. Insure that flow through the dry gas meter is less than 1% of the sampling rate.

3.2 Connect the probe and sample at a constant rate of 0.075 c.f.m. or at a rate proportional to the stack gas velocity. Continue sampling until the dry gas meter registers 1 cubic foot or until visible liquid droplets are carried over from the first impinger to the second. Record temperature, pressure, and dry gas meter readings as required by Figure 4-2.

3.3 After collecting the sample, measure the volume increase to the nearest 0.5 ml.

4. Calculations.

4.1 Volume of water vapor collected.

$$V_{wc}=\frac{(V_f-V_i)\rho_{H_2O}RT_{std}}{P_{std}M_{H_2O}}=0.0474\frac{ft.^3}{ml.}(V_f-V_i)$$

equation 4-1

where:

$V_{wc}$ = Volume of water vapor collected (standard conditions), cu. ft.

$V_f$ = Final volume of impinger contents, ml.

$V_i$ = Initial volume of impinger contents, ml.

R = Ideal gas constant, 21.83 inches Hg—cu. ft./lb. mole-°R.

$\rho_{H_2O}$ = Density of water, 1 g./ml.

$T_{std}$ = Absolute temperature at standard conditions, 530° R.

$P_{std}$ = Absolute pressure at standard conditions, 29.92 inches Hg.

$M_{H_2O}$ = Molecular weight of water, 18 lb./lb.-mole.

4.2 Gas volume.

$$V_{mc}=V_m\left(\frac{P_m}{P_{std}}\right)\left(\frac{T_{std}}{T_m}\right)=17.71\frac{°R}{in.\ Hg}\left(\frac{V_mP_m}{T_m}\right)$$

equation 4-2

where:

$V_{mc}$ = Dry gas volume through meter at standard conditions, cu. ft.

$V_m$ = Dry gas volume measured by meter, cu. ft.

$P_m$ = Barometric pressure at the dry gas meter, inches Hg.

$P_{std}$ = Pressure at standard conditions, 29.92 inches Hg.

$T_{std}$ = Absolute temperature at standard conditions, 530° R.

$T_m$ = Absolute temperature at meter (°F + 460), °R.

4.3 Moisture content.

$$B_{wo}=\frac{V_{wc}}{V_{wc}+V_{mc}}+B_{wm}=\frac{V_{wc}}{V_{wc}+V_{mc}}+(0.025)$$

equation 4-3

where:

$B_{wo}$ = Proportion by volume of water vapor in the gas stream, dimensionless.

$V_{wc}$ = Volume of water vapor collected (standard conditions), cu. ft.

$V_{mc}$ = Dry gas volume through meter (standard conditions), cu. ft.

$B_{wm}$ = Approximate volumetric proportion of water vapor in the gas stream leaving the impingers, 0.025.

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[1] If liquid droplets are present in the gas stream, assume the stream to be saturated, determine the average stack gas temperature by traversing according to Method 1, and use a psychrometric chart to obtain an approximation of the moisture percentage.

[2] Trade name.

6. References

Air Pollution Engineering Manual, Danielson, J. A. (ed.), U.S. DHEW, PHS, National Center for Air Pollution Control, Cincinnati, Ohio, PHS Publication No. 999-AP-40, 1967.

Devorkin, Howard, et al., Air Pollution Source Testing Manual, Air Pollution Control District, Los Angeles, Calif., November 1963.

Methods for Determination of Velocity, Volume, Dust and Mist Content of Gases, Western Precipitation Division of Joy Manufacturing Co., Los Angeles, Calif., Bulletin WP-50, 1968.

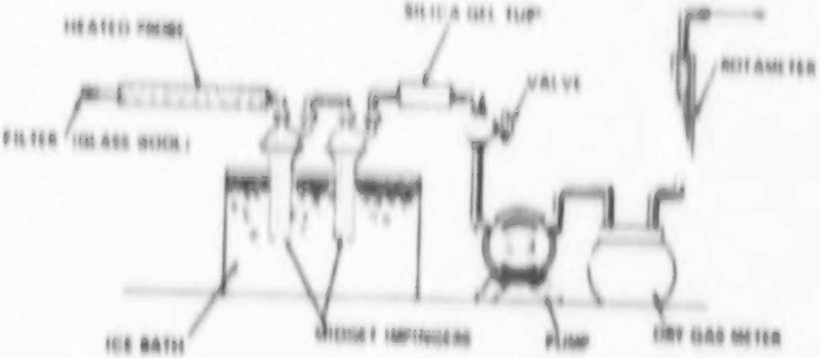
HEATED PROBE
SILICA GEL TUBE
ROTAMETER
VALVE
FILTER (GLASS WOOL)
ICE BATH
MIDGET IMPINGERS
PUMP
DRY GAS METER

Figure 4-1. Moisture-sampling train.

LOCATION ______________________ COMMENTS
TEST ______________________
DATE ______________________
OPERATOR ______________________

BAROMETRIC PRESSURE ______________________

| CLOCK TIME | GAS VOLUME THROUGH METER, (Vm), ft³ | ROTAMETER SETTING ft³/min | METER TEMPERATURE °F |
|---|---|---|---|
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |

Figure 4-2. Field moisture determination.

# METHOD 5

# Determination Of Particulate Emissions From Stationary Sources

1. *Principle and applicability.*

1.1 Principle. Particulate matter is withdrawn isokinetically from the source and its weight is determined gravimetrically after removal of uncombined water.

1.2 Applicability. This method is applicable for the determination of particulate emissions from stationary sources only when specified by the test procedures for determining compliance with New Source Performance Standards.

2. *Apparatus.*

2.1 Sampling train. The design specifications of the particulate sampling train used by EPA (Figure 5-1) are described in APTD-0581. Commercial models of this train are available.

2.1.1 Nozzle—Stainless steel (316) with sharp, tapered leading edge.

2.1.2 Probe—Pyrex[1] glass with a heating system capable of maintaining a minimum gas temperature of 250° F. at the exit end during sampling to prevent condensation from occurring. When length limitations (greater than about 8 ft.) are encountered at temperatures less than 600° F., Incoloy 825[1], or equivalent, may be used. Probes for sampling gas streams at temperatures in excess of 600° F. must have been approved by the Administrator.

2.1.3 Pitot tube—Type S, or equivalent, attached to probe to monitor stack gas velocity.

2.1.4 Filter Holder—Pyrex[1] glass with heating system capable of maintaining minimum temperature of 225° F.

2.1.5 Impingers / Condenser—Four impingers connected in series with glass ball joint fittings. The first, third, and fourth impingers are of the Greenburg-Smith design, modified by replacing the tip with a ½-inch ID glass tube extending to one-half inch from the bottom of the flask. The second impinger is of the Greenburg-Smith design with the standard tip. A condenser may be used in place of the impingers provided that the moisture content of the stack gas can still be determined.

2.1.6 Metering system—Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 5° F., dry gas meter with 2% accuracy, and related equipment, or equivalent, as required to maintain an isokinetic sampling rate and to determine sample volume.

2.1.7 Barometer—To measure atmospheric pressure to ±0.1 inches Hg.

2.2 Sample recovery.

2.2.1 Probe brush—At least as long as probe.

2.2.2 Glass wash bottles—Two.

2.2.3 Glass sample storage containers.

2.2.4 Graduated cylinder—250 ml.

2.3 Analysis.

2.3.1 Glass weighing dishes.

2.3.2 Desiccator.

2.3.3 Analytical balance—To measure to 0.1 mg.

2.3.4 Trip balance—300 g. capacity, to measure to ±0.05 g.

3. *Reagents.*

3.1 Sampling.

3.1.1 Filters—Glass fiber, MSA 1106 BH[1], or equivalent, numbered for identification and preweighed.

3.1.2 Silica gel—Indicating type, 6-16 mesh, dried at 175° C. (350° F.) for 2 hours.

3.1.3 Water.

3.1.4 Crushed ice.

3.2 Sample recovery.

3.2.1 Acetone—Reagent grade.

3.3 Analysis.

3.3.1 Water.

3.3.2 Desiccant—Drierite[1], indicating.

4. *Procedure.*

4.1 Sampling.

4.1.1 After selecting the sampling site and the minimum number of sampling points, determine the stack pressure, temperature, moisture, and range of velocity head.

4.1.2 Preparation of collection train. Weigh to the nearest gram approximately 200 g. of silica gel. Label a filter of proper diameter, desiccate[2] for at least 24 hours and weigh to the nearest 0.5 mg. in a room where the relative humidity is less than 50%. Place 100 ml. of water in each of the first two impingers, leave the third impinger empty and place approximately 200 g. of preweighed silica gel in the fourth impinger. Set up the train without the probe as in Figure 5-1. Leak check the sampling train at the sampling site by plugging up the inlet to the filter holder and pulling a 15 in. Hg vacuum. A leakage rate not in excess of 0.02 c.f.m. at a vacuum of 15 in. Hg is acceptable. Attach the probe and adjust the heater to provide a gas temperature of about 250° F. at the probe outlet. Turn on the filter heating system. Place crushed ice around the impingers. Add more ice during the run to keep the temperature of the gases leaving the last impinger as low as possible and preferably at 70° F., or less. Temperatures above 70° F. may result in damage to the dry gas meter from either moisture condensation or excessive heat.

4.1.3 Particulate train operation. For each run, record the data required on the example sheet shown in Figure 5-2. Take readings at each sampling point, at least every 5 minutes, and when significant changes in stack flow conditions necessitate additional adjustments in flow rate. To begin sampling, position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Sample for at least 5 minutes at each traverse point; sampling time must be the same for each point. Maintain isokinetic sampling throughout the sampling period. Nomographs are available which aid in the rapid adjustment of the sampling rate without other computations. APTD-0576 details the procedure for using these nomographs. Turn off the pump at the conclusion of each run and record the final readings. Remove the probe and nozzle from the stack and handle in accordance with the sample recovery process described in section 4.2.

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[1] Trade name.

[2] Dry using Drierite[1] at 70° F ±10° F.

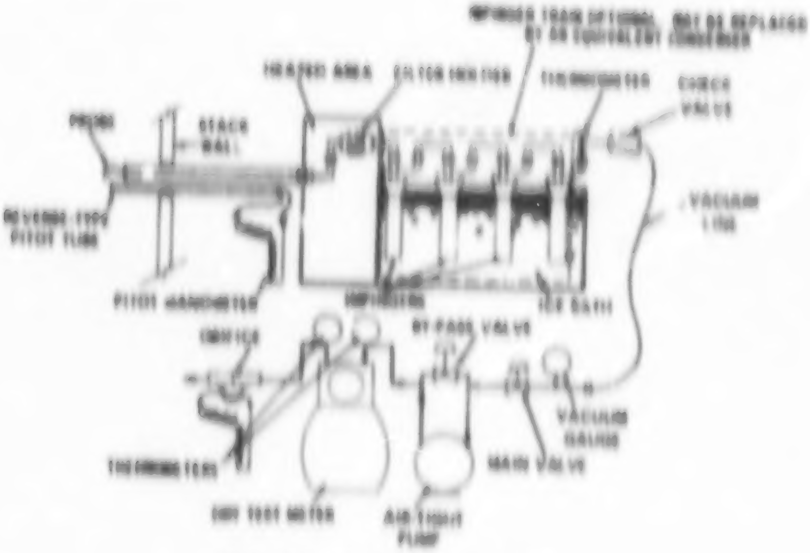
IMPINGER TRAIN OPTIONAL, MAY BE REPLACED BY AN EQUIVALENT CONDENSER
HEATED AREA
FILTER HOLDER
THERMOMETER
CHECK VALVE
PROBE
STACK WALL
REVERSE-TYPE PITOT TUBE
VACUUM LINE
PITOT MANOMETER
IMPINGERS
ICE BATH
BY-PASS VALVE
ORIFICE
VACUUM GAUGE
THERMOMETERS
MAIN VALVE
DRY TEST METER
AIR-TIGHT PUMP

Figure 5-1. Particulate sampling train.

Figure 5-2. Particulate field data.

4.3 Sample recovery. Exercise care in moving the collection train from the test site to the sample recovery area to minimize the loss of collected sample or the gain of extraneous particulate matter. Set aside a portion of the acetone used in the sample recovery as a blank for analysis. Measure the volume of water from the first three impingers, then discard. Place the samples in containers as follows:

Container No. 1. Remove the filter from its holder, place in this container, and seal.

Container No. 2. Place loose particulate matter and acetone washings from all sample-exposed surfaces prior to the filter in this container and seal. Use a razor blade, brush, or rubber policeman to lose adhering particles.

Container No. 3. Transfer the silica gel from the fourth impinger to the original container and seal. Use a rubber policeman as an aid in removing silica gel from the impinger.

4.4 Analysis. Record the data required on the example sheet shown in Figure 5-3. Handle each sample container as follows:

Container No. 1. Transfer the filter and any loose particulate matter from the sample container to a tared glass weighing dish, desiccate, and dry to a constant weight. Report results to the nearest 0.5 mg.

Container No. 2. Transfer the acetone washings to a tared beaker and evaporate to dryness at ambient temperature and pressure. Desiccate and dry to a constant weight. Report results to the nearest 0.5 mg.

Container No. 3. Weigh the spent silica gel and report to the nearest gram.

5. Calibration.

Use methods and equipment which have been approved by the Administrator to calibrate the orifice meter, pitot tube, dry gas meter, and probe heater. Recalibrate after each test series.

6. Calculations.

6.1 Average dry gas meter temperature and average orifice pressure drop. See data sheet (Figure 5-3).

6.2 Dry gas volume. Correct the sample volume measured by the dry gas meter to standard conditions (70° F., 29.92 inches Hg) by using equation 5-1.

$$V_{m_{std}} = V_m\left(\frac{T_{std}}{T_m}\right)\left(\frac{P_{bar}+\frac{\Delta H}{13.6}}{P_{std}}\right) = \left(17.71\ \frac{°R}{\text{in. Hg}}\right)V_m\left(\frac{P_{bar}+\frac{\Delta H}{13.6}}{T_m}\right)$$

equation 5-1

where:

$V_{m_{std}}$ = Volume of gas sample through the dry gas meter (standard conditions), cu. ft.

$V_m$ = Volume of gas sample through the dry gas meter (meter conditions), cu. ft.

$T_{std}$ = Absolute temperature at standard conditions, 530° R.

$T_m$ = Average dry gas meter temperature, °R.

$P_{bar}$ = Barometric pressure at the orifice meter, inches Hg.

$\Delta H$ = Average pressure drop across the orifice meter, inches $H_2O$.

13.6 = Specific gravity of mercury.

$P_{std}$ = Absolute pressure at standard conditions, 29.92 inches Hg.

6.3 Volume of water vapor.

$$V_{w_{std}} = V_{l_c}\left(\frac{\rho_{H_2O}}{M_{H_2O}}\right)\left(\frac{R\,T_{std}}{P_{std}}\right) = \left(0.0474\ \frac{\text{cu. ft.}}{\text{ml.}}\right)V_{l_c}$$

equation 5-2

where:

$V_{w_{std}}$ = Volume of water vapor in the gas sample (standard conditions), cu. ft.

$V_{l_c}$ = Total volume of liquid collected in impingers and silica gel (see Figure 5-3), ml.

$\rho_{H_2O}$ = Density of water, 1 g./ml.

$M_{H_2O}$ = Molecular weight of water, 18 lb./lb.-mole.

$R$ = Ideal gas constant, 21.83 inches Hg—cu. ft./lb.-mole-°R.

$T_{std}$ = Absolute temperature at standard conditions, 530° R.

$P_{std}$ = Absolute pressure at standard conditions, 29.92 inches Hg.

6.4 Moisture content.

$$B_{wo} = \frac{V_{w_{std}}}{V_{m_{std}}+V_{w_{std}}}$$

equation 5-3

where:

$B_{wo}$ = Proportion by volume of water vapor in the gas stream, dimensionless.

$V_{w_{std}}$ = Volume of water in the gas sample (standard conditions), cu. ft.

$V_{m_{std}}$ = Volume of gas through the dry gas meter (standard conditions), cu. ft.

6.5 Total particulate weight. Determine the total particulate catch from the sum of the weights on the analysis data sheet (Figure 5-3).

6.6 Concentration.

6.6.1 Concentration in gr./s.c.f.

$$c'_s = \left(0.0154\ \frac{\text{gr.}}{\text{mg.}}\right)\left(\frac{M_n}{V_{m_{std}}}\right)$$

equation 5-4

where:

$c'_s$ = Concentration of particulate matter in stack gas, dry basis, corrected to standard conditions, gr./s.c.f.

$M_n$ = Total amount of particulate matter collected, mg.

$V_{m_{std}}$ = Volume of gas sample through dry gas meter (standard conditions), cu. ft.

6.6.2 Concentration in g./cu. m.

$$c_s = \frac{\left(\frac{0.001\ \text{g.}}{\text{mg.}}\right)M_n}{V_{m_{std}}\ 0.0283\ \frac{\text{m.}^3}{\text{ft.}^3}} = 0.0353\ \frac{M_n}{V_{m_{std}}}$$

equation 5-5

where:

$c_s$ = Concentration of particulate matter in stack gas, dry basis, corrected to standard conditions, g./cu. m.

$M_n$ = Total amount of particulate matter collected, mg.

$V_{m_{std}}$ = Volume of gas sample through dry gas meter (standard conditions), cu. ft.

PLANT ____________________
DATE ____________________
RUN NO. ____________________

| CONTAINER NUMBER | WEIGHT OF PARTICULATE COLLECTED, mg | | |
|---|---|---|---|
| | FINAL WEIGHT | TARE WEIGHT | WEIGHT GAIN |
| 1 | | | |
| 2 | | | |
| TOTAL | | | |

| | VOLUME OF LIQUID WATER COLLECTED | | |
|---|---|---|---|
| | IMPINGER VOLUME, ml | SILICA GEL WEIGHT, g | |
| FINAL | | | |
| INITIAL | | | |
| LIQUID COLLECTED | | | |
| TOTAL VOLUME COLLECTED | | g* | ml |

*CONVERT WEIGHT OF WATER TO VOLUME BY DIVIDING TOTAL WEIGHT INCREASE BY DENSITY OF WATER. (1 g/ml)

$$\frac{\text{INCREASE, g}}{(1\ \text{g/ml})} = \text{VOLUME WATER, ml}$$

Figure 5-3. Analytical data.

[illegible]

$$[illegible]$$

[illegible]

[illegible]

[illegible]

[illegible]

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# METHOD 6

## Determination Of Sulfur Dioxide Emissions From Stationary Sources

1. Principle and applicability.

1.1 Principle. A gas sample is extracted from the sampling point in the stack. The acid mist, including sulfur trioxide, is separated from the sulfur dioxide. The sulfur dioxide fraction is measured by the barium-thorin titration method.

1.2 Applicability. This method is applicable for the determination of sulfur dioxide emissions from stationary sources only when specified by the test procedures for determining compliance with New Source Performance Standards.

2. Apparatus.

2.1 Sampling. See Figure 6-1.

2.1.1 Probe—Pyrex[1] glass, approximately 5 to 6 mm. ID, with a heating system to prevent condensation and a filtering medium to remove particulate matter including sulfuric acid mist.

2.1.2 Midget bubbler—One, with glass wool packed in top to prevent sulfuric acid mist carryover.

2.1.3 Glass wool.

2.1.4 Midget impingers—Three.

2.1.5 Drying tube—Packed with 6 to 16 mesh indicating-type silica gel, or equivalent, to dry the sample.

2.1.6 Valve—Needle valve, or equivalent, to adjust flow rate.

2.1.7 Pump—Leak-free, vacuum type.

2.1.8 Rate meter—Rotameter or equivalent, to measure a 0-10 s.c.f.h. flow range.

2.1.9 Dry gas meter—Sufficiently accurate to measure the sample volume within 1%.

2.1.10 Pitot tube—Type S, or equivalent, necessary only if a sample traverse is required, or if stack gas velocity varies with time.

2.2 Sample recovery.

2.2.1 Glass wash bottles—Two.

2.2.2 Polyethylene storage bottles—To store impinger samples.

2.3 Analysis.

2.3.1 Pipettes—Transfer type, 5 ml. and 10 ml. sizes (0.1 ml. divisions) and 25 ml. size (0.2 ml. divisions).

2.3.2 Volumetric flasks—50 ml., 100 ml., and 1,000 ml.

2.3.3 Burettes—5 ml. and 50 ml.

2.3.4 Erlenmeyer flask—125 ml.

3. Reagents.

3.1 Sampling.

3.1.1 Water—Deionized, distilled.

3.1.2 Isopropanol, 80%—Mix 80 ml. of isopropanol with 20 ml. of distilled water.

3.1.3 Hydrogen peroxide, 3%—Dilute 100 ml. of 30% hydrogen peroxide to 1 liter with distilled water. Prepare fresh daily.

3.2 Sample recovery.

3.2.1 Water—Deionized, distilled.

3.2.2 Isopropanol, 80%.

3.3 Analysis.

3.3.1 Water—Deionized, distilled.

3.3.2 Isopropanol.

3.3.3 Thorin indicator—1-(o-arsonophenylazo)-2-naphthol-3,6-disulfonic acid, disodium salt (or equivalent). Dissolve 0.20 g. in 100 ml. distilled water.

3.3.4 Barium perchlorate (0.01 N)—Dissolve 1.95 g. of barium perchlorate [$Ba(ClO_4)_2 \cdot 3H_2O$] in 200 ml. distilled water and dilute to 1 liter with isopropanol. Barium chloride may be used.

3.3.5 Sulfuric acid standard (0.01 N)—Purchase or standardize to ±0.0002 N against 0.01 N NaOH which has previously been standardized against primary standard potassium acid phthalate.

4. Procedure.

4.1 Sampling.

4.1.1 Preparation of collection train. Pour 15 ml. of 80% isopropanol into the midget bubbler and 15 ml. of 3% hydrogen peroxide into each of the first two midget impingers. Leave the final midget impinger dry. Assemble the train as shown in Figure 6-1. Leak check the sampling train at the sampling site by plugging the probe inlet and pulling a 10 inches Hg vacuum. A leakage rate not in excess of 1% of the sampling rate is acceptable. Carefully release the probe inlet plug and turn off the pump. Place crushed ice around the impingers. Add more ice during the run to keep the temperature of the gases leaving the last impinger at 70° F. or less.

4.1.2 Sample collection. Adjust the sample flow rate proportional to the stack gas velocity. Take readings at least every five minutes and when significant changes in stack conditions necessitate additional adjustments in flow rate. To begin sampling, position the tip of the probe at the first sampling point and start the pump. Sample proportionally throughout the run. At the conclusion of each run, turn off the pump and record the final readings. Remove the probe from the stack and disconnect it from the train. Drain the ice bath and purge the remaining part of the train by drawing clean ambient air through the system for 15 minutes.

4.2 Sample recovery. Disconnect the impingers after purging. Discard the contents of the midget bubbler. Pour the contents of the midget impingers into a polyethylene shipment bottle. Rinse the three midget impingers and the connecting tubes with distilled water and add these washings to the same storage container.

4.3 Sample analysis. Transfer the contents of the storage container to a 50 ml. volumetric flask. Dilute to the mark with deionized, distilled water. Pipette a 10 ml. aliquot of this solution into a 125 ml. Erlenmeyer flask. Add 40 ml. of isopropanol and two to four drops of thorin indicator. Titrate to a pink endpoint using 0.01 N barium perchlorate. Run a blank with each series of samples.

5. Calibration.

5.1 Use standard methods and equipment which have been approved by the Administrator to calibrate the rotameter, pitot tube, dry gas meter, and probe heater.

---

[1] Trade name.

5.5 Standardize the barium perchlorate against 25 ml. of standard sulfuric acid containing 100 ml. of isopropanol.

6. Calculations.

6.1 Dry gas volume. Correct the sample volume measured by the dry gas meter to standard conditions (70° F. and 29.92 inches Hg) by using equation 6-1.

$$V_{m_{std}} = V_m \left(\frac{T_{std}}{T_m}\right)\left(\frac{P_{bar}}{P_{std}}\right) = 17.71 \frac{°R}{in.\ Hg}\left(\frac{V_m P_{bar}}{T_m}\right) \quad \text{equation 6-1}$$

where:

$V_{m_{std}}$ = Volume of gas sample through the dry gas meter (standard conditions), cu. ft.

$V_m$ = Volume of gas sample through the dry gas meter (meter conditions), cu. ft.

$T_{std}$ = Absolute temperature at standard conditions, 530° R.

$T_m$ = Average dry gas meter temperature, °R.

$P_{bar}$ = Barometric pressure at the orifice meter, inches Hg.

$P_{std}$ = Absolute pressure at standard conditions, 29.92 inches Hg.

6.2 Sulfur dioxide concentration.

$$C_{SO_2} = \left(7.05 \times 10^{-5} \frac{lb.-l.}{g.-ml.}\right) \frac{(V_t - V_{tb}) N \left(\frac{V_{soln}}{V_a}\right)}{V_{m_{std}}} \quad \text{equation 6-2}$$

where:

$C_{SO_2}$ = Concentration of sulfur dioxide at standard conditions, dry basis, lb./cu. ft.

$7.05 \times 10^{-5}$ = Conversion factor, including the number of grams per gram equivalent of sulfur dioxide (32 g./g.-eq.), 453.6 g./lb., and 1,000 ml./l., lb.-l./g.-ml.

$V_t$ = Volume of barium perchlorate titrant used for the sample, ml.

$V_{tb}$ = Volume of barium perchlorate titrant used for the blank, ml.

$N$ = Normality of barium perchlorate titrant, g.-eq./l.

$V_{soln}$ = Total solution volume of sulfur dioxide, 50 ml.

$V_a$ = Volume of sample aliquot titrated, ml.

$V_{m_{std}}$ = Volume of gas sample through the dry gas meter (standard conditions), cu. ft., see Equation 6-1.

7. References.

Atmospheric Emissions from Sulfuric Acid Manufacturing Processes, U.S. DHEW, PHS, Division of Air Pollution, Public Health Service Publication No. 999-AP-13, Cincinnati, Ohio, 1965.

Corbett, P. F., The Determination of $SO_2$ and $SO_3$ in Flue Gases, Journal of the Institute of Fuel, 24:237-243, 1961.

Matty, R. E. and E. K. Diehl, Measuring Flue-Gas $SO_2$ and $SO_3$, Power 101:94-97, November, 1957.

Patton, W. F. and J. A. Brink, Jr., New Equipment and Techniques for Sampling Chemical Process Gases, J. Air Pollution Control Association, 13, 162 (1963).

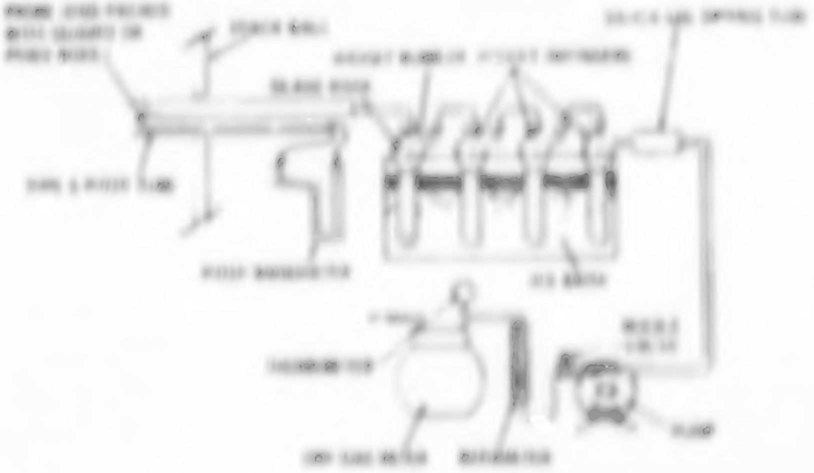

Figure 6-1. $SO_2$ sampling train.

# METHOD 7

## Determination Of Nitrogen Oxide Emissions From Stationary Sources

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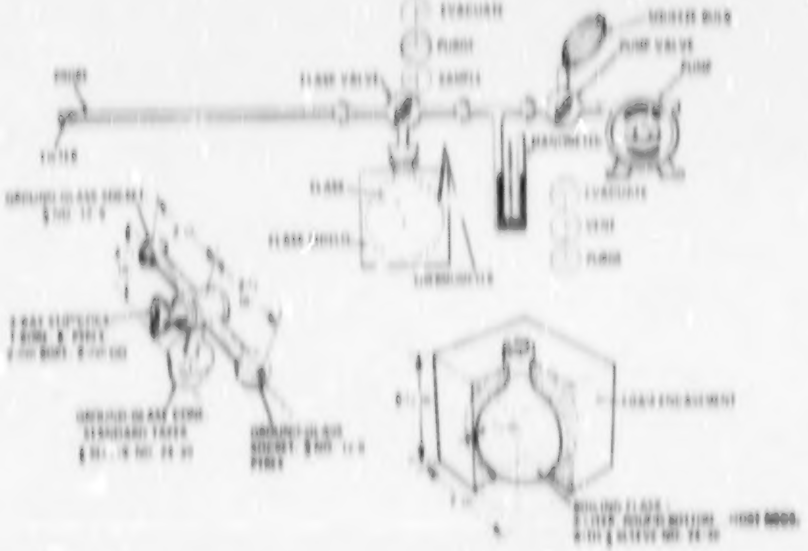
EVACUATE
PURGE
SAMPLE
SQUEEZE BULB
PUMP VALVE
PUMP
PROBE
FLASK VALVE
FILTER
MANOMETER
FLASK
FLASK SHIELD
THERMOMETER
EVACUATE
VENT
PURGE
GROUND-GLASS SOCKET § NO. 12/5
3-WAY STOPCOCK T-BORE, § PYREX 2-mm BORE, 8-mm OD
GROUND-GLASS CONE STANDARD TAPER § SLEEVE NO. 24/40
GROUND-GLASS SOCKET, § NO. 12/5 PYREX
FOAM ENCASEMENT
BOILING FLASK - 2-LITER, ROUND-BOTTOM, SHORT NECK, WITH § SLEEVE NO. 24/40

Figure F-1. Sampling train, flask valve, and flask.

in the sample. For a blank use 25 ml. of absorbing solution and the same volume of distilled water as used in diluting the flask. Prior to shipping or analysis, add sodium hydroxide (1N) dropwise into both the sample and the blank until alkaline to litmus paper (about 25 to 35 drops in each).

4.3 Analysis.

4.3.1 If the sample has been shipped in a container, transfer the contents to a 250 ml. beaker using a small amount of distilled water. Evaporate the solution to dryness on a steam bath and then cool. Add 2 ml. phenoldisulfonic acid solution to the dried residue and triturate thoroughly with a glass rod. Make sure the solution contacts all the residue. Add 1 ml. distilled water and four drops of concentrated sulfuric acid. Heat the solution on a steam bath for 3 minutes with occasional stirring. Cool, add 20 ml. distilled water, mix well by stirring, and add concentrated ammonium hydroxide dropwise with constant stirring until alkaline to litmus paper. Transfer the solution to a 100 ml. volumetric flask and wash the beaker three times with 4 to 5 ml. portions of distilled water. Dilute to the mark and mix thoroughly. If the sample contains solids, transfer a portion of the solution to a clean, dry centrifuge tube, and centrifuge, or filter a portion of the solution. Measure the absorbance of each sample at 420 nm. using the blank solution as a zero. Dilute the sample and the blank with a suitable amount of distilled water if absorbance falls outside the range of calibration.

5. Calibration.

5.1 Flask volume. Assemble the flask and flask valve and fill with water to the stopcock. Measure the volume of water to ±10 ml. Number and record the volume on the flask.

5.2 Spectrophotometer. Add 0.0 to 16.0 ml. of standard solution to a series of beakers. To each beaker add 25 ml. of absorbing solution and add sodium hydroxide (1N) dropwise until alkaline to litmus paper (about 25 to 35 drops). Follow the analysis procedure of section 4.3 to collect enough data to draw a calibration curve of concentration in μg. $NO_2$ per sample versus absorbance.

6. Calculations.

6.1 Sample volume.

$$V_{sc}=\frac{T_{std}(V_f-V_a)}{P_{std}}\left(\frac{P_f}{T_f}-\frac{P_i}{T_i}\right)=\left(17.71\ \frac{^\circ R}{\text{in. Hg}}\right)(V_f-25\ \text{ml.})\left(\frac{P_f}{T_f}-\frac{P_i}{T_i}\right)\qquad \text{Equation 7-1}$$

where:

$V_{sc}$ = Sample volume at standard conditions (dry basis), ml.

$T_{std}$ = Absolute temperature at standard conditions, 530° R.

$P_{std}$ = Pressure at standard conditions, 29.92 inches Hg.

$V_f$ = Volume of flask and valve, ml.

$V_a$ = Volume of absorbing solution, 25 ml.

$P_f$ = Final absolute pressure of flask, inches Hg.

$P_i$ = Initial absolute pressure of flask, inches Hg.

$T_f$ = Final absolute temperature of flask, °R.

$T_i$ = Initial absolute temperature of flask, °R.

6.2 Sample concentration. Read μg. $NO_2$ for each sample from the plot of μg. $NO_2$ versus absorbance.

$$C=\left(\frac{m}{V_{sc}}\right)\left(\frac{\frac{1\ \text{lb.}}{\text{cu. ft.}}}{1.6\times10^{10}\frac{\mu\text{g.}}{\text{ml.}}}\right)=\left(6.2\times10^{-11}\frac{\text{lb./s.c.f.}}{\mu\text{g./ml.}}\right)\left(\frac{m}{V_{sc}}\right)$$

Equation 7-2

where:

C = Concentration of $NO_x$ as $NO_2$ (dry basis), lb./s.c.f.

m = Mass of $NO_2$ in gas sample, μg.

$V_{sc}$ = Sample volume at standard conditions (dry basis), ml.

7. *References.*

Standard Methods of Chemical Analysis. 6th ed. New York, D. Van Nostrand Co., Inc., 1962, vol. 1, p. 329-330.

Standard Method of Test for Oxides of Nitrogen in Gaseous Combustion Products (Phenoldisulfonic Acid Procedure). In: 1968 Book of ASTM Standards, Part 26, Philadelphia, Pa. 1968. ASTM Designation D-1608-60, p. 725-729.

Jacob, M. B. The Chemical Analysis of Air Pollutants. New York, N.Y. Interscience Publishers, Inc. 1960, vol. 10, p. 351-356.

# METHOD 8

# Determination Of Sulfuric Acid Mist And Sulfur Dioxide Emissions From Stationary Sources

1. *Principle and applicability.*

1.1 Principle. A gas sample is extracted from a sampling point in the stack and the acid mist including sulfur trioxide is separated from sulfur dioxide. Both fractions are measured separately by the barium-thorin titration method.

1.2 Applicability. This method is applicable to determination of sulfuric acid mist (including sulfur trioxide) and sulfur dioxide from stationary sources only when specified by the test procedures for determining compliance with the New Source Performance Standards.

2. Apparatus.

2.1 Sampling. See Figure 8-1. Many of the design specifications of this sampling train are described in APTD-0581.

2.1.1 Nozzle—Stainless steel (316) with sharp, tapered leading edge.

2.1.2 Probe—Pyrex[1] glass with a heating system to prevent visible condensation during sampling.

2.1.3 Pitot tube—Type S, or equivalent, attached to probe to monitor stack gas velocity.

2.1.4 Filter holder—Pyrex[1] glass.

2.1.5 Impingers—Four as shown in Figure 8-1. The first and third are of the Greenburg-Smith design with standard tip. The second and fourth are of the Greenburg-Smith design, modified by replacing the standard tip with a ½-inch ID glass tube extending to one-half inch from the bottom of the impinger flask. Similar collection systems, which have been approved by the Administrator, may be used.

2.1.6 Metering system—Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 5° F., dry gas meter with 2% accuracy, and related equipment, or equivalent, as required to maintain an isokinetic sampling rate and to determine sample volume.

2.1.7 Barometer—To measure atmospheric pressure to ±0.1 inch Hg.

2.2 Sample recovery.

2.2.1 Wash bottles—Two.

2.2.2 Graduated cylinders—250 ml., 500 ml.

2.2.3 Glass sample storage containers.

2.2.4 Graduated cylinder—250 ml.

2.3 Analysis.

2.3.1 Pipette—25 ml., 100 ml.

2.3.2 Burette—50 ml.

2.3.3 Erlenmeyer flask—250 ml.

2.3.4 Graduated cylinder—100 ml.

2.3.5 Trip balance—300 g. capacity, to measure to ±0.05 g.

2.3.6 Dropping bottle—to add indicator solution.

3. Reagents.

3.1 Sampling.

3.1.1 Filters—Glass fiber, MSA type 1106 BH, or equivalent, of a suitable size to fit in the filter holder.

3.1.2 Silica gel—Indicating type, 6-16 mesh, dried at 175° C. (350° F.) for 2 hours.

3.1.3 Water—Deionized, distilled.

3.1.4 Isopropanol, 80%—Mix 800 ml. of isopropanol with 200 ml. of deionized, distilled water.

3.1.5 Hydrogen peroxide, 3%—Dilute 100 ml. of 30% hydrogen peroxide to 1 liter with deionized, distilled water.

3.1.6 Crushed ice.

3.2 Sample recovery.

3.2.1 Water—Deionized, distilled.

3.2.2 Isopropanol, 80%.

3.3 Analysis.

3.3.1 Water—Deionized, distilled.

3.3.2 Isopropanol.

3.3.3 Thorin indicator—1-(o-arsonophenylazo)-2-naphthol-3,6-disulfonic acid, disodium salt (or equivalent). Dissolve 0.20 g. in 100 ml. distilled water.

3.3.4 Barium perchlorate (0.01N)—Dissolve 1.95 g. of barium perchlorate [$Ba(ClO_4)_2 \cdot 3H_2O$] in 200 ml. distilled water and dilute to 1 liter with isopropanol. Standardize with sulfuric acid.

3.3.5 Sulfuric acid standard (0.01N)—Purchase or standardize to ±0.0002 N against 0.01N NaOH which has previously been standardized against primary standard potassium acid phthalate.

4. Procedure.

4.1 Sampling.

4.1.1 After selecting the sampling site and the minimum number of sampling points, determine the stack pressure, temperature, moisture, and range of velocity head.

4.1.2 Preparation of collection train. Place 100 ml. of 80% isopropanol in the first impinger, 100 ml. of 3% hydrogen peroxide in both the second and third impingers, and about 200 g. of silica gel in the fourth impinger. Retain a portion of the reagents for use as blank solutions. Assemble the train without the probe as shown in Figure 8-1 with the filter between the first and second impingers. Leak check the sampling train at the sampling site by plugging the inlet to the first impinger and pulling a 15-inch Hg vacuum. A leakage rate not in excess of 0.02 c.f.m. at a vacuum of 15 inches Hg is acceptable. Attach the probe and turn on the probe heating system. Adjust the probe heater setting during sampling to prevent any visible condensation. Place crushed ice around the impingers. Add more ice during the run to keep the temperature of the gases leaving the last impinger at 70° F. or less.

4.1.3 Train operation. For each run, record the data required on the example sheet shown in Figure 8-2. Take readings at each sampling point at least every 5 minutes and when significant changes in stack conditions necessitate additional adjustments in flow rate. To begin sampling, position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Start the pump and immediately adjust the flow to isokinetic conditions. Maintain isokinetic sampling throughout the sampling period. Nomographs are available which aid in the rapid adjustment of the sampling rate without other computations. APTD-0576 details the procedure for using the nomographs.

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[1] Trade name.

At the conclusion of each run, turn off the pump and record the final readings. Remove the probe from the stack and disconnect it from the glass tubing. Drain the ice bath and purge the remaining part of the train by drawing clean ambient air through the system for 15 minutes.

6.2 Sample recovery.

6.2.1 Transfer the isopropanol from the first impinger to a 250 ml. graduated cylinder. Rinse the probe, first impinger, and all connecting glassware before the filter with 80% isopropanol. Add the rinse solution to the cylinder. Dilute to 250 ml. with 80% isopropanol. Add the filter to the solution, mix, and transfer to a suitable storage container. Transfer the solution from the second and third impingers to a 500 ml. graduated cylinder. Rinse all glassware between the filter and silica gel impinger with distilled water and add this rinse water to the cylinder. Dilute to a volume of 500 ml. with deionized, distilled water. Transfer the solution to a suitable storage container.

6.3 Analysis.

6.3.1 Shake the container holding isopropanol and the filter. If the filter breaks up, allow the fragments to settle for a few minutes before removing a sample. Pipette a 100 ml. aliquot of sample into a 250 ml. Erlenmeyer flask and add 2 to 4 drops of thorin indicator. Titrate the sample with barium perchlorate to a pink end point. Make sure to record volumes. Repeat the titration with a second aliquot of sample. Shake the container holding the contents of the second and third impingers. Pipette a 10 ml. aliquot of sample into a 250 ml. Erlenmeyer flask. Add 40 ml. of isopropanol and 2 to 4 drops of thorin indicator. Titrate the sample with barium perchlorate to a pink end point. Repeat the titration with a second aliquot of sample. Titrate the blanks in the same manner as the samples.

7. Calibration.

7.1 Use standard methods and equipment which have been approved by the Administrator to calibrate the orifice meter, pitot tube, dry gas meter, and probe heater.

7.2 Standardize the barium perchlorate with 25 ml. of standard sulfuric acid, containing 100 ml. of isopropanol.

8. Calculations.

8.1 Dry gas volume. Correct the sample volume measured by the dry gas meter to standard conditions (70° F., 29.92 inches Hg) by using Equation 8-1.

$$V_{m_{std}} = V_m \frac{T_{std}}{T_m}\left(\frac{P_{bar}+\frac{\Delta H}{13.6}}{P_{std}}\right) = \left(17.71\frac{°R}{in.\ Hg}\right)V_m\left(\frac{P_{bar}+\frac{\Delta H}{13.6}}{T_m}\right)$$

Equation 8-1

where:

$V_{m_{std}}$ = Volume of gas sample through the dry gas meter (standard conditions), cu. ft.

$T_{std}$ = Absolute temperature at standard conditions, 530° R.

$T_m$ = Average dry gas meter temperature, °R.

$P_{bar}$ = Barometric pressure at the orifice meter, inches Hg.

$V_m$ = Volume of gas sample through the dry gas meter (meter conditions), cu. ft.

$\Delta H$ = Pressure drop across the orifice meter, inches $H_2O$.

13.6 = Specific gravity of mercury.

$P_{std}$ = Absolute pressure at standard conditions, 29.92 inches Hg.

8.2 Sulfuric acid concentration.

$$C_{H_2SO_4} = \left(1.08\times10^{-4}\frac{lb.\text{-}l.}{g.\text{-}ml.}\right)\frac{(V_t-V_{tb})(N)\left(\frac{V_{soln}}{V_a}\right)}{V_{m_{std}}}$$

Equation 8-2

$C_{H_2SO_4}$ = Concentration of sulfuric acid at standard conditions, dry basis, lb./cu. ft.

$1.08\times10^{-4}$ = Conversion factor including the number of grams per gram equivalent of sulfuric acid (49 g./g.-eq.), 453.6 g./lb., and 1,000 ml./l., lb.-l./g.-ml.

$V_t$ = Volume of barium perchlorate titrant used for the sample, ml.

$V_{tb}$ = Volume of barium perchlorate titrant used for the blank, ml.

$N$ = Normality of barium perchlorate titrant, g.-eq./l.

$V_{soln}$ = Total solution volume of sulfuric acid (first impinger and filter), ml.

$V_a$ = Volume of sample aliquot titrated, ml.

$V_{m_{std}}$ = Volume of gas sample through the dry gas meter (standard conditions), cu. ft., see Equation 8-1.

8.3 Sulfur dioxide concentration.

$$C_{SO_2} = \left(7.05\times10^{-5}\frac{lb.\text{-}l.}{g.\text{-}ml.}\right)\frac{(V_t-V_{tb})(N)\left(\frac{V_{soln}}{V_a}\right)}{V_{m_{std}}}$$

Equation 8-3

where:

$C_{SO_2}$ = Concentration of sulfur dioxide at standard conditions, dry basis, lb./cu. ft.

$7.05\times10^{-5}$ = Conversion factor including the number of grams per gram equivalent of sulfur dioxide (32 g./g.-eq.), 453.6 g./lb., and 1,000 ml./l., lb.-l./g.-ml.

$V_t$ = Volume of barium perchlorate titrant used for the sample, ml.

$V_{tb}$ = Volume of barium perchlorate titrant used for the blank, ml.

$N$ = Normality of barium perchlorate titrant, g.-eq./l.

$V_{soln}$ = Total solution volume of sulfur dioxide (second and third impingers), ml.

$V_a$ = Volume of sample aliquot titrated, ml.

$V_{m_{std}}$ = Volume of gas sample through the dry gas meter (standard conditions), cu. ft., see Equation 8-1.

## 9. References

Atmospheric Emissions from Sulfuric Acid Manufacturing Processes, U.S. DHEW, PHS, Division of Air Pollution, Public Health Service Publication No. 999-AP-13, Cincinnati, Ohio, 1965.

Corbett, P. F., The Determination of SO2 and SO3 in Flue Gases, Journal of the Institute of Fuel, 24:237-243, 1961.

Martin, Robert M., Construction Details of Isokinetic Source Sampling Equipment, Environmental Protection Agency, Air Pollution Control Office Publication No. APTD-0581.

Patton, W. F. and J. A. Brink, Jr., New Equipment and Techniques for Sampling Chemical Process Gases, J. Air Pollution Control Assoc. 13, 162 (1963).

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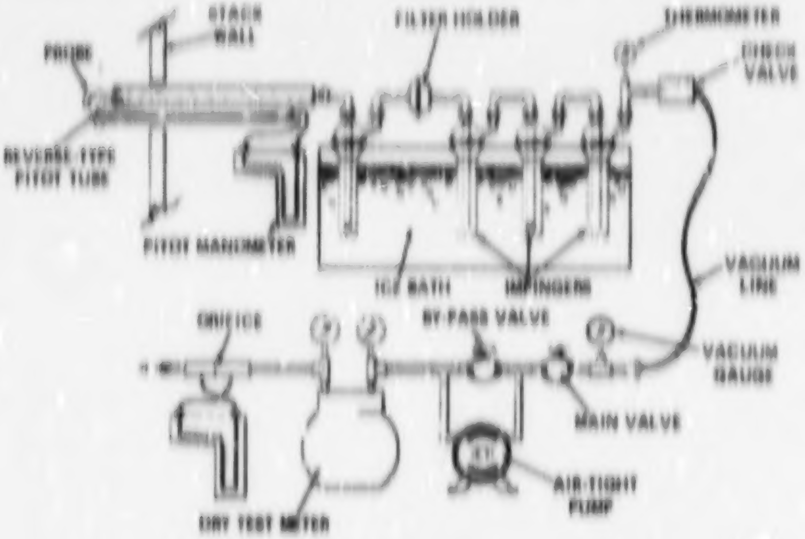
STACK WALL
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PROBE
CHECK VALVE
REVERSE-TYPE PITOT TUBE
PITOT MANOMETER
ICE BATH
IMPINGERS
VACUUM LINE
ORIFICE
BY-PASS VALVE
VACUUM GAUGE
MAIN VALVE
AIR-TIGHT PUMP
DRY TEST METER

Figure 8-1. Sulfuric acid mist sampling train.

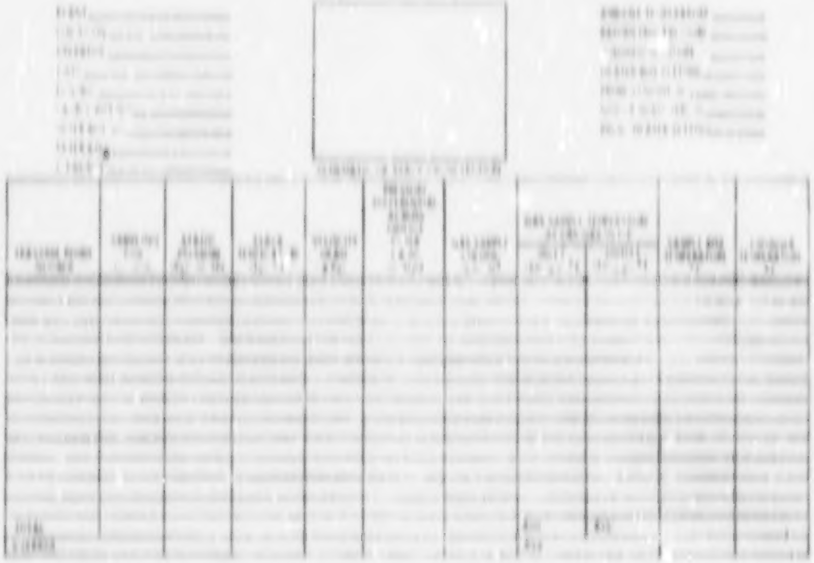

Figure 8-2. Field data.

# METHOD 9

## Visual Determination Of The Opacity Of Emissions From Stationary Sources

Many stationary sources discharge visible emissions into the atmosphere; these emissions are usually in the shape of a plume. This method involves the determination of plume opacity by qualified observers. The method includes procedures for the training and certification of observers, and procedures to be used in the field for determination of plume opacity. The appearance of a plume as viewed by an observer depends upon a number of variables, some of which may be controllable and some of which may not be controllable in the field. Variables which can be controlled to an extent to which they no longer exert a significant influence upon plume appearance include: Angle of the observer with respect to the plume; angle of the observer with respect to the sun; point of observation of attached and detached steam plume; and angle of the observer with respect to a plume emitted from a rectangular stack with a large length to width ratio. The method includes specific criteria applicable to these variables.

Other variables which may not be controllable in the field are luminescence and color contrast between the plume and the background against which the plume is viewed. These variables exert an influence upon the appearance of a plume as viewed by an observer, and can affect the ability of the observer to accurately assign opacity values to the observed plume. Studies of the theory of plume opacity and field studies have demonstrated that a plume is most visible and presents the greatest apparent opacity when viewed against a contrasting background. It follows from this, and is confirmed by field trials, that the opacity of a plume, viewed under conditions where a contrasting background is present can be assigned with the greatest degree of accuracy. However, the potential for a positive error is also the greatest when a plume is viewed under such contrasting conditions. Under conditions presenting a less contrasting background, the apparent opacity of a plume is less and approaches zero as the color and luminescence contrast decrease toward zero. As a result, significant negative bias and negative errors can be made when a plume is viewed under less contrasting conditions. A negative bias decreases rather than increases the possibility that a plant operator will be cited for a violation of opacity standards due to observer error.

Studies have been undertaken to determine the magnitude of positive errors which can be made by qualified observers while reading plumes under contrasting conditions and using the procedures set forth in this method. The results of these studies (field trials) which involve a total of 769 sets of 25 readings each are as follows:

(1) For black plumes (133 sets at a smoke generator), 100 percent of the sets were read with a positive error[1] of less than 7.5 percent opacity; 99 percent were read with a positive error of less than 5 percent opacity.

(2) For white plumes (170 sets at a smoke generator, 168 sets at a coal-fired power plant, 298 sets at a sulfuric acid plant), 99 percent of the sets were read with a positive error of less than 7.5 percent opacity; 95 percent were read with a positive error of less than 5 percent opacity.

The positive observational error associated with an average of twenty-five readings is therefore established. The accuracy of the method must be taken into account when determining possible violations of applicable opacity standards.

1. *Principle and applicability.*

1.1 *Principle.* The opacity of emissions from stationary sources is determined visually by a qualified observer.

1.2 *Applicability.* This method is applicable for the determination of the opacity of emissions from stationary sources pursuant to § 60.11(b) and for qualifying observers for visually determining opacity of emissions.

2. *Procedures.* The observer qualified in accordance with paragraph 3 of this method shall use the following procedures for visually determining the opacity of emissions:

2.1 *Position.* The qualified observer shall stand at a distance sufficient to provide a clear view of the emissions with the sun oriented in the 140° sector to his back. Consistent with maintaining the above requirement, the observer shall, as much as possible, make his observations from a position such that his line of vision is approximately perpendicular to the plume direction, and when observing opacity of emissions from rectangular outlets (e.g. roof monitors, open baghouses, noncircular stacks), approximately perpendicular to the longer axis of the outlet. The observer's line of sight should not include more than one plume at a time when multiple stacks are involved, and in any case the observer should make his observations with his line of sight perpendicular to the longer axis of such a set of multiple stacks (e.g. stub stacks on baghouses).

2.2 *Field records.* The observer shall record the name of the plant, emission location, type facility, observer's name and affiliation, and the date on a field data sheet (Figure 9-1). The time, estimated distance to the emission location, approximate wind direction, estimated wind speed, description of the sky condition (presence and color of clouds), and plume background are recorded on a field data sheet at the time opacity readings are initiated and completed.

2.3 *Observations.* Opacity observations shall be made at the point of greatest opacity in that portion of the plume where condensed water vapor is not present. The observer shall not look continuously at the plume, but instead shall observe the plume momentarily at 15-second intervals.

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[1] For a set, positive error = average opacity determined by observers' 25 observations − average opacity determined from transmissometer's 25 recordings.

2.3.1 Attached steam plumes. When condensed water vapor is present within the plume as it emerges from the emission outlet, opacity observations shall be made beyond the point in the plume at which condensed water vapor is no longer visible. The observer shall record the approximate distance from the emission outlet to the point in the plume at which the observations are made.

2.3.2 Detached steam plume. When water vapor in the plume condenses and becomes visible at a distinct distance from the emission outlet, the opacity of emissions should be evaluated at the emission outlet prior to the condensation of water vapor and the formation of the steam plume.

2.4 Recording observations. Opacity observations shall be recorded to the nearest 5 percent at 15-second intervals on an observational record sheet. (See Figure 9-2 for an example.) A minimum of 24 observations shall be recorded. Each momentary observation recorded shall be deemed to represent the average opacity of emissions for a 15-second period.

2.5 Data Reduction. Opacity shall be determined as an average of 24 consecutive observations recorded at 15-second intervals. Divide the observations recorded on the record sheet into sets of 24 consecutive observations. A set is composed of any 24 consecutive observations. Sets need not be consecutive in time and in no case shall two sets overlap. For each set of 24 observations, calculate the average by summing the opacity of the 24 observations and dividing this sum by 24. If an applicable standard specifies an averaging time requiring more than 24 observations, calculate the average for all observations made during the specified time period. Record the average opacity on a record sheet. (See Figure 9-1 for an example.)

3. Qualifications and testing.

3.1 Certification requirements. To receive certification as a qualified observer, a candidate must be tested and demonstrate the ability to assign opacity readings in 5 percent increments to 25 different black plumes and 25 different white plumes, with an error not to exceed 15 percent opacity on any one reading and an average error not to exceed 7.5 percent opacity in each category. Candidates shall be tested according to the procedures described in paragraph 3.2. Smoke generators used pursuant to paragraph 3.2 shall be equipped with a smoke meter which meets the requirements of paragraph 3.3.

The certification shall be valid for a period of 6 months, at which time the qualification procedure must be repeated by any observer in order to retain certification.

3.2 Certification procedure. The certification test consists of showing the candidate a complete run of 50 plumes—25 black plumes and 25 white plumes—generated by a smoke generator. Plumes within each set of 25 black and 25 white runs shall be presented in random order. The candidate assigns an opacity value to each plume and records his observation on a suitable form. At the completion of each run of 50 readings, the score of the candidate is determined. If a candidate fails to qualify, the complete run of 50 readings must be repeated in any retest. The smoke test may be administered as part of a smoke school or training program, and may be preceded by training or familiarization runs of the smoke generator during which candidates are shown black and white plumes of known opacity.

3.3 Smoke generator specifications. Any smoke generator used for the purposes of paragraph 3.2 shall be equipped with a smoke meter installed to measure opacity across the diameter of the smoke generator stack. The smoke meter output shall display in-stack opacity based upon a pathlength equal to the stack exit diameter, on a full 0 to 100 percent chart recorder scale. The smoke meter optical design and performance shall meet the specifications shown in Table 9-1. The smoke meter shall be calibrated as prescribed in paragraph 3.3.1 prior to the conduct of each smoke reading test. At the completion of each test, the zero and span drift shall be checked and if the drift exceeds ±1 percent opacity, the condition shall be corrected prior to conducting any subsequent test runs. The smoke meter shall be demonstrated, at the time of installation, to meet the specifications listed in Table 9-1. This demonstration shall be repeated following any subsequent repair or replacement of the photocell or associated electronic circuitry including the chart recorder or output meter, or every 6 months, whichever occurs first.

TABLE 9-1.—SMOKE METER DESIGN AND PERFORMANCE SPECIFICATIONS

| Parameter: | Specification |
|---|---|
| a. Light source | Incandescent lamp operated at nominal rated voltage. |
| b. Spectral response of photocell. | Photopic (daylight spectral response of the human eye—reference 4.3). |
| c. Angle of view | 15° maximum total angle. |
| d. Angle of projection. | 15° maximum total angle. |
| e. Calibration error. | ±3% opacity, maximum. |
| f. Zero and span drift. | ±1% opacity, 30 minutes. |
| g. Response time | ≤5 seconds. |

3.3.1 Calibration. The smoke meter is calibrated after allowing a minimum of 30 minutes warmup by alternately producing simulated opacity of 0 percent and 100 percent. When stable response at 0 percent or 100 percent is noted, the smoke meter is adjusted to produce an output of 0 percent or 100 percent, as appropriate. This calibration shall be repeated until stable 0 percent and 100 percent readings are produced without adjustment. Simulated 0 percent and 100 percent opacity values may be produced by alternately switching the power to the light source on and off while the smoke generator is not producing smoke.

3.3.2 Smoke meter evaluation. The smoke meter design and performance are to be evaluated as follows:

3.3.2.1 Light source. Verify from manufacturer's data and from voltage measurements made at the lamp, as installed, that the lamp is operated within ±5 percent of the nominal rated voltage.

3.3.2.2 Spectral response of photocell. Verify from manufacturer's data that the photocell has a photopic response; i.e., the spectral sensitivity of the cell shall closely approximate the standard spectral-luminosity curve for photopic vision which is referenced in (b) of Table 9-1.

3.2.3 **Angle of view.** Check construction geometry to ensure that the total angle of view of the smoke plume, as seen by the photocell, does not exceed 15°. The total angle of view may be calculated from: θ=2 tan⁻¹ d/2L, where θ= total angle of view; d= the sum of the photocell diameter + the diameter of the limiting aperture; and L= the distance from the photocell to the limiting aperture. The limiting aperture is the point in the path between the photocell and the smoke plume where the angle of view is most restricted. In smoke generator smoke meters this is normally an orifice plate.

3.2.4 **Angle of projection.** Check construction geometry to ensure that the total angle of projection of the lamp on the smoke plume does not exceed 15°. The total angle of projection may be calculated from: θ=2 tan⁻¹ d/2L, where θ= total angle of projection; d= the sum of the length of the lamp filament + the diameter of the limiting aperture; and L= the distance from the lamp to the limiting aperture.

3.2.5 **Calibration error.** Using neutral-density filters of known opacity, check the error between the actual response and the theoretical linear response of the smoke meter. This check is accomplished by first calibrating the smoke meter according to 3.3.1 and then inserting a series of three neutral-density filters of nominal opacity of 20, 50, and 75 percent in the smoke meter pathlength. Filters calibrated within ±2 percent shall be used. Care should be taken when inserting the filters to prevent stray light from affecting the meter. Make a total of five nonconsecutive readings for each filter. The maximum error on any one reading shall be 3 percent opacity.

3.2.6 **Zero and span drift.** Determine the zero and span drift by calibrating and operating the smoke generator in a normal manner over a 1-hour period. The drift is measured by checking the zero and span at the end of this period.

3.2.7 **Response time.** Determine the response time by producing the series of five simulated 0 percent and 100 percent opacity values and observing the time required to reach stable response. Opacity values of 0 percent and 100 percent may be simulated by alternately switching the power to the light source off and on while the smoke generator is not operating.

4. *References.*

4.1 Air Pollution Control District Rules and Regulations, Los Angeles County Air Pollution Control District, Regulation IV, Prohibitions, Rule 50.

4.2 Weisburd, Melvin I., Field Operations and Enforcement Manual for Air, U.S. Environmental Protection Agency, Research Triangle Park, N.C., APTD-1100, August 1972, pp. 4.1-4.36.

4.3 Condon, E.U., and Odishaw, H., Handbook of Physics, McGraw-Hill Co., N.Y., N.Y., 1958, Table 3.1, p. 6-52.

FIGURE 9-1
RECORD OF VISUAL DETERMINATION OF OPACITY

PAGE___of___

COMPANY________
LOCATION________
TEST NUMBER________
DATE________
TYPE FACILITY________
CONTROL DEVICE________

HOURS OF OBSERVATION________
OBSERVER________
OBSERVER CERTIFICATION DATE________
OBSERVER AFFILIATION________
POINT OF EMISSIONS________
HEIGHT OF DISCHARGE POINT________

| | Initial | | | Final |
|---|---|---|---|---|
| CLOCK TIME | | | | |
| OBSERVER LOCATION<br>Distance to Discharge | | | | |
| Direction from Discharge | | | | |
| Height of Observation Point | | | | |
| BACKGROUND DESCRIPTION | | | | |
| WEATHER CONDITIONS<br>Wind Direction | | | | |
| Wind Speed | | | | |
| Ambient Temperature | | | | |
| SKY CONDITIONS (clear, overcast, % clouds, etc.) | | | | |
| PLUME DESCRIPTION<br>Color | | | | |
| Distance Visible | | | | |
| OTHER INFORMATION | | | | |

SUMMARY OF AVERAGE OPACITY

| Set Number | Time<br>Start—End | Opacity<br>Sum | Average |
|---|---|---|---|
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Readings ranged from ___ to ___ % opacity

The source was/was not in compliance with ___ at the time evaluation was made.

Reproduced from best available copy.

FIGURE 9-2 OBSERVATION RECORD PAGE ___ OF ___

COMPANY ____________________
LOCATION ____________________
TEST NUMBER ____________________
DATE ____________________

OBSERVER ____________________
TYPE FACILITY ____________________
POINT OF EMISSIONS ____________________

|  |  | Seconds |  |  |  | STEAM PLUME (check if applicable) |  |  |
|---|---|---|---|---|---|---|---|---|
| Hr. | Min. | 0 | 15 | 30 | 45 | Attached | Detached | COMMENTS |
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### FIGURE 9-2 OBSERVATION RECORD (Continued)

PAGE ___ OF ___

| | |
|---|---|
| COMPANY ________ | OBSERVER ________ |
| LOCATION ________ | TYPE FACILITY ________ |
| TEST NUMBER ________ | POINT OF EMISSIONS ________ |
| DATE ________ | |

| Hr. | Min. | Seconds 0 | 15 | 30 | 45 | STEAM PLUME (check if applicable) Attached | Detached | COMMENTS |
|---|---|---|---|---|---|---|---|---|
| | 30 | | | | | | | |
| | 31 | | | | | | | |
| | 32 | | | | | | | |
| | 33 | | | | | | | |
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# METHOD 10

## Determination Of Carbon Monoxide Emissions From Stationary Sources

1. *Principle and Applicability.*

1.1 *Principle.* An integrated or continuous gas sample is extracted from a sampling point and analyzed for carbon monoxide (CO) content using a Luft-type nondispersive infrared analyzer (NDIR) or equivalent.

1.2 *Applicability.* This method is applicable for the determination of carbon monoxide emissions from stationary sources only when specified by the test procedures for determining compliance with new source performance standards. The test procedure will indicate whether a continuous or an integrated sample is to be used.

2. *Range and sensitivity.*

2.1 *Range.* 0 to 1,000 ppm.

2.2 *Sensitivity.* Minimum detectable concentration is 20 ppm for a 0 to 1,000 ppm span.

3. *Interferences.* Any substance having a strong absorption of infrared energy will interfere to some extent. For example, discrimination ratios for water ($H_2O$) and carbon dioxide ($CO_2$) are 3.5 percent $H_2O$ per 7 ppm CO and 10 percent $CO_2$ per 10 ppm CO, respectively, for devices measuring in the 1,500 to 3,000 ppm range. For devices measuring in the 0 to 100 ppm range, interference ratios can be as high as 3.5 percent $H_2O$ per 25 ppm CO and 10 percent $CO_2$ per 50 ppm CO. The use of silica gel and ascarite traps will alleviate the major interference problems. The measured gas volume must be corrected if these traps are used.

4. *Precision and accuracy.*

4.1 *Precision.* The precision of most NDIR analyzers is approximately ±2 percent of span.

4.2 *Accuracy.* The accuracy of most NDIR analyzers is approximately ±5 percent of span after calibration.

5. *Apparatus.*

5.1 *Continuous sample (Figure 10-1).*

5.1.1 *Probe.* Stainless steel or sheathed Pyrex[1] glass, equipped with a filter to remove particulate matter.

5.1.2 *Air-cooled condenser or equivalent.* To remove any excess moisture.

5.2 *Integrated sample (Figure 10-2).*

5.2.1 *Probe.* Stainless steel or sheathed Pyrex glass, equipped with a filter to remove particulate matter.

5.2.2 *Air-cooled condenser or equivalent.* To remove any excess moisture.

5.2.3 *Valve.* Needle valve, or equivalent, to adjust flow rate.

5.2.4 *Pump.* Leak-free diaphragm type, or equivalent, to transport gas.

5.2.5 *Rate meter.* Rotameter, or equivalent, to measure a flow range from 0 to 1.0 liter per min. (0.035 cfm).

5.2.6 *Flexible bag.* Tedlar, or equivalent, with a capacity of 60 to 90 liters (2 to 3 ft³). Leak-test the bag in the laboratory before using by evacuating bag with a pump followed by a dry gas meter. When evacuation is complete, there should be no flow through the meter.

5.2.7 *Pitot tube.* Type S, or equivalent, attached to the probe so that the sampling rate can be regulated proportional to the stack gas velocity when velocity is varying with the time or a sample traverse is conducted.

5.3 *Analysis (Figure 10-3).*

5.3.1 *Carbon monoxide analyzer.* Nondispersive infrared spectrometer, or equivalent. This instrument should be demonstrated, preferably by the manufacturer, to meet or exceed manufacturer's specifications and those described in this method.

5.3.2 *Drying tube.* To contain approximately 200 g of silica gel.

5.3.3 *Calibration gas.* Refer to paragraph 6.1.

5.3.4 *Filter.* As recommended by NDIR manufacturer.

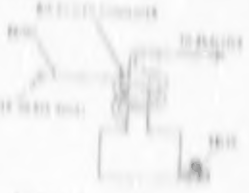

Figure 10-1. Continuous sampling train.

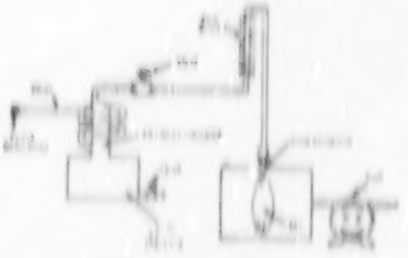

Figure 10-2. Integrated gas-sampling train.

5.3.5 *CO₂ removal tube.* To contain approximately 500 g of ascarite.

5.3.6 *Ice water bath.* For ascarite and silica gel tubes.

5.3.7 *Valve.* Needle valve, or equivalent, to adjust flow rate.

5.3.8 *Rate meter.* Rotameter or equivalent to measure gas flow rate of 0 to 1.0 liter per min. (0.035 cfm) through NDIR.

5.3.9 *Recorder (optional).* To provide permanent record of NDIR readings.

6. *Reagents.*

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[1] Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

6.1 *Calibration gases.* Known concentration of CO in nitrogen ($N_2$) for instrument span, prepurified grade of $N_2$ for zero, and two additional concentrations corresponding approximately to 60 percent and 30 percent span. The span concentration shall not exceed 1.5 times the applicable source performance standard. The calibration gases shall be certified by the manufacturer to be within ±2 percent of the specified concentration.

FIGURE 10-3.—Analytical equipment.

6.2 *Silica gel.* Indicating type, 6 to 16 mesh, dried at 175° C (347° F) for 2 hours.

6.3 *Ascarite.* Commercially available.

7. *Procedure.*

7.1 *Sampling.*

7.1.1 *Continuous sampling.* Set up the equipment as shown in Figure 10-1 making sure all connections are leak free. Place the probe in the stack at a sampling point and purge the sampling line. Connect the analyzer and begin drawing sample into the analyzer. Allow 5 minutes for the system to stabilize, then record the analyzer reading as required by the test procedure. (See 7.2 and 8). $CO_2$ content of the gas may be determined by using the Method 3 integrated sample procedure (36 FR 24886), or by weighing the ascarite $CO_2$ removal tube and computing $CO_2$ concentration from the gas volume sampled and the weight gain of the tube.

7.1.2 *Integrated sampling.* Evacuate the flexible bag. Set up the equipment as shown in Figure 10-2 with the bag disconnected. Place the probe in the stack and purge the sampling line. Connect the bag, making sure that all connections are leak free. Sample at a rate proportional to the stack velocity. $CO_2$ content of the gas may be determined by using the Method 3 integrated sample procedures (36 FR 24886), or by weighing the ascarite $CO_2$ removal tube and computing $CO_2$ concentration from the gas volume sampled and the weight gain of the tube.

7.2 *CO Analysis.* Assemble the apparatus as shown in Figure 10-3, calibrate the instrument, and perform other required operations as described in paragraph 8. Purge analyzer with $N_2$ prior to introduction of each sample. Direct the sample stream through the instrument for the test period, recording the readings. Check the zero and span again after the test to assure that any drift or malfunction is detected. Record the sample data on Table 10-1.

8. *Calibration.* Assemble the apparatus according to Figure 10-3. Generally an instrument requires a warm-up period before stability is obtained. Follow the manufacturer's instructions for specific procedure. Allow a minimum time of one hour for warm-up. During this time check the sample conditioning apparatus, i.e., filter, condenser, drying tube, and $CO_2$ removal tube, to ensure that each component is in good operating condition. Zero and calibrate the instrument according to the manufacturer's procedures using, respectively, nitrogen and the calibration gases.

9. *Calculation—Concentration of carbon monoxide.* Calculate the concentration of carbon monoxide in the stack using equation 10-1.

$$C_{CO_{stack}}=C_{CO_{NDIR}}(1-F_{CO_2}) \qquad \text{equation 10-1}$$

where:

$C_{CO_{stack}}$ = concentration of CO in stack, ppm by volume (dry basis).

$C_{CO_{NDIR}}$ = concentration of CO measured by NDIR analyzer, ppm by volume (dry basis).

$F_{CO_2}$ = volume fraction of $CO_2$ in sample, i.e., percent $CO_2$ from Orsat analysis divided by 100.

10. *Bibliography.*

10.1 McElroy, Frank, The Intertech NDIR-CO Analyzer, Presented at 11th Methods Conference on Air Pollution, University of California, Berkeley, Calif., April 1, 1970.

10.2 Jacobs, M. B., et al., Continuous Determination of Carbon Monoxide and Hydrocarbons in Air by a Modified Infrared Analyzer, J. Air Pollution Control Association, 9(2):110-114, August 1959.

10.3 MSA LIRA Infrared Gas and Liquid Analyzer Instruction Book, Mine Safety Appliances Co., Technical Products Division, Pittsburgh, Pa.

10.4 Models 215A, 315A, and 415A Infrared Analyzers, Beckman Instruments, Inc., Beckman Instructions 1635-B, Fullerton, Calif., October 1967.

10.5 Continuous CO Monitoring System, Model A5611, Intertech Corp., Princeton, N.J.

10.6 UNOR Infrared Gas Analyzers, Bendix Corp., Ronceverte, West Virginia.

TABLE 10-1.—*Field data*

**Location** ..................................................... **Comments:**
**Test** .....................................................
**Date** .....................................................
**Operator** .....................................................

| *Clock time* | *Rotameter setting, liters per minute (cubic feet per minute)* |
|---|---|
| | |

## Addenda

*A. Performance Specifications for NDIR Carbon Monoxide Analyzers.*

| | |
|---|---|
| Range (minimum) | 0–100 ppm. |
| Output (minimum) | 0–10 mV. |
| Minimum detectable sensitivity | 0.5 ppm. |
| Rise time, 90 percent (maximum) | 30 seconds. |
| Fall time, 90 percent (maximum) | 30 seconds. |
| Zero drift (maximum) | 10% in 8 hours. |
| Span drift (maximum) | 10% in 8 hours. |
| Precision (minimum) | ± 2% of full scale. |
| Noise (maximum) | ± 1% of full scale. |
| Linearity (maximum deviation) | 2% of full scale. |
| Interference rejection ratio | $CO_2$—1000 to 1, $H_2O$—500 to 1. |

*B. Definitions of Performance Specifications.*

*Range*—The minimum and maximum measurement limits.

*Output*—Electrical signal which is proportional to the measurement; intended for connection to readout or data processing devices. Usually expressed as millivolts or milliamps full scale at a given impedance.

*Full scale*—The maximum measuring limit for a given range.

*Minimum detectable sensitivity*—The smallest amount of input concentration that can be detected as the concentration approaches zero.

*Accuracy*—The degree of agreement between a measured value and the true value; usually expressed as ± percent of full scale.

*Time to 90 percent response*—The time interval from a step change in the input concentration at the instrument inlet to a reading of 90 percent of the ultimate recorded concentration.

*Rise Time (90 percent)*—The interval between initial response time and time to 90 percent response after a step increase in the inlet concentration.

*Fall Time (90 percent)*—The interval between initial response time and time to 90 percent response after a step decrease in the inlet concentration.

*Zero Drift*—The change in instrument output over a stated time period, usually 24 hours, of unadjusted continuous operation when the input concentration is zero; usually expressed as percent full scale.

*Span Drift*—The change in instrument output over a stated time period, usually 24 hours, of unadjusted continuous operation when the input concentration is a stated upscale value; usually expressed as percent full scale.

*Precision*—The degree of agreement between repeated measurements of the same concentration, expressed as the average deviation of the single results from the mean.

*Noise*—Spontaneous deviations from a mean output not caused by input concentration changes.

*Linearity*—The maximum deviation between an actual instrument reading and the reading predicted by a straight line drawn between upper and lower calibration points.

# METHOD 11

# Determination Of Hydrogen Sulfide Emissions From Stationary Sources

1. *Principle and applicability.*

1.1 *Principle.* Hydrogen sulfide ($H_2S$) is collected from the source in a series of midget impingers and reacted with alkaline cadmium hydroxide [$Cd(OH)_2$] to form cadmium sulfide (CdS). The precipitated CdS is then dissolved in hydrochloric acid and absorbed in a known volume of iodine solution. The iodine consumed is a measure of the $H_2S$ content of the gas. An impinger containing hydrogen peroxide is included to remove $SO_2$ as an interfering species.

1.2 *Applicability.* This method is applicable for the determination of hydrogen sulfide emissions from stationary sources only when specified by the test procedures for determining compliance with the new source performance standards.

2. *Apparatus.*

2.1 *Sampling train.*

2.1.1 *Sampling line*—6- to 7-mm (¼-inch) Teflon[1] tubing to connect sampling train to sampling valve, with provisions for heating to prevent condensation. A pressure reducing valve prior to the Teflon sampling line may be required depending on sampling stream pressure.

2.1.2 *Impingers*—Five midget impingers, each with 30-ml capacity, or equivalent.

2.1.3 *Ice bath container*—To maintain absorbing solution at a constant temperature.

2.1.4 *Silica gel drying tube*—To protect pump and dry gas meter.

2.1.5 *Needle valve, or equivalent*—Stainless steel or other corrosion resistant material, to adjust gas flow rate.

2.1.6 *Pump*—Leak free, diaphragm type, or equivalent, to transport gas. (Not required if sampling stream under positive pressure.)

2.1.7 *Dry gas meter*—Sufficiently accurate to measure sample volume to within 1 percent.

2.1.8 *Rate meter*—Rotameter, or equivalent, to measure a flow rate of 0 to 3 liters per minute (0.1 $ft^3$/min).

2.1.9 *Graduated cylinder*—25 ml.

2.1.10 *Barometer*—To measure atmospheric pressure within ±2.5 mm (0.1 in.) Hg.

2.2 *Sample Recovery.*

2.2.1 *Sample container*—500-ml glass-stoppered iodine flask.

2.2.2 *Pipette*—50-ml volumetric type.

2.2.3 *Beakers*—250 ml.

2.2.4 *Wash bottle*—Glass.

2.3 *Analysis.*

2.3.1 *Flask*—500-ml glass-stoppered iodine flask.

2.3.2 *Burette*—One 50 ml.

2.3.3 *Flask*—125-ml conical.

3. *Reagents.*

3.1 *Sampling.*

3.1.1 *Absorbing solution*—Cadmium hydroxide ($Cd(OH)_2$)—Mix 4.3 g cadmium sulfate hydrate ($3CdSO_4 \cdot 8H_2O$) and 0.3 g of sodium hydroxide (NaOH) in 1 liter of distilled water ($H_2O$). Mix well.

*Note:* The cadmium hydroxide formed in this mixture will precipitate as a white suspension. Therefore, this solution must be thoroughly mixed before using to ensure an even distribution of the cadmium hydroxide.

3.1.2 *Hydrogen peroxide, 3 percent*—Dilute 30 percent hydrogen peroxide to 3 percent as needed. Prepare fresh daily.

3.2 *Sample recovery.*

3.2.1 *Hydrochloric acid solution (HCl), 10 percent by weight*—Mix 230 ml of concentrated HCl (specific gravity 1.19) and 770 ml of distilled $H_2O$.

3.2.2 *Iodine solution, 0.1 N*—Dissolve 24 g potassium iodide (KI) in 30 ml of distilled $H_2O$ in a 1-liter graduated cylinder. Weigh 12.7 g of resublimed iodine ($I_2$) into a weighing bottle and add to the potassium iodide solution. Shake the mixture until the iodine is completely dissolved. Slowly dilute the solution to 1 liter with distilled $H_2O$, with swirling. Filter the solution, if cloudy, and store in a brown glass-stoppered bottle.

3.2.3 *Standard iodine solution, 0.01 N*—Dilute 100 ml of the 0.1 N iodine solution in a volumetric flask to 1 liter with distilled water.

Standardize daily as follows: Pipette 25 ml of the 0.01 N iodine solution into a 125-ml conical flask. Titrate with standard 0.01 N thiosulfate solution (see paragraph 3.3.2) until the solution is a light yellow. Add a few drops of the starch solution and continue titrating until the blue color just disappears. From the results of this titration, calculate the exact normality of the iodine solution (see paragraph 5.1).

3.2.4 *Distilled, deionized water.*

3.3 *Analysis.*

3.3.1 *Sodium thiosulfate solution, standard 0.1 N*—For each liter of solution, dissolve 24.8 g of sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$) in distilled water and add 0.01 g of anhydrous sodium carbonate ($Na_2CO_3$) and 0.4 ml of chloroform ($CHCl_3$) to stabilize. Mix thoroughly by shaking or by aerating with nitrogen for approximately 15 minutes, and store in a glass-stoppered glass bottle.

Standardize frequently as follows: Weigh into a 500-ml volumetric flask about 2 g of potassium dichromate ($K_2Cr_2O_7$) weighed to the nearest milligram and dilute to the 500-ml mark with distilled $H_2O$. Use dichromate which has been crystallized from distilled water and oven-dried at 182°C to 199°C (360°F to 390°F). Dissolve approximately 3 g of potassium iodide (KI) in 50 ml of distilled water in a glass-stoppered, 500-ml conical flask, then add 5 ml of 20-percent hydrochloric acid solution. Pipette 50 ml of the dichromate solution into this mixture. Gently swirl the solution once and allow it to stand in the dark for 5 minutes. Dilute the solution with 100 to 200 ml of distilled water, washing down the sides of the flask with part of the water. Swirl the solution slowly and titrate with the thiosulfate solution until the solution is light yellow. Add 4 ml of starch solution and continue with a titrate with the thiosulfate until the bright blue color has disappeared and only the pale green color of the chromic ion remains. From this titration, calculate the exact normality of the sodium thiosulfate solution (see paragraph 5.2).

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[1] Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

3.3.6 Sodium thiosulfate solution, standard 0.01 N. Pipette 100 ml of the standard 0.1 N thiosulfate solution into a volumetric flask and dilute to one liter with distilled water.

3.3.7 Starch indicator solution. Suspend 10 g of soluble starch in 100 ml of distilled water and add 15 g of potassium hydroxide pellets. Stir until dissolved, dilute with 900 ml of distilled water and let stand for 1 hour. Neutralize the alkali with concentrated hydrochloric acid, using an indicator paper similar to Alkacid test ribbon, then add 2 ml of glacial acetic acid as a preservative.

Test for decomposition by titrating 4 ml of starch solution in 200 ml of distilled water with 0.01 N iodine solution. If more than 4 drops of the 0.01 N iodine solution are required to obtain the blue color, make up a fresh starch solution.

4. Procedure.

4.1 Sampling.

4.1.1 Assemble the sampling train as shown in Figure 11-1, connecting the five midget impingers in series. Place 15 ml of 3 percent hydrogen peroxide in the first impinger. Place 15 ml of the absorbing solution in each of the next three impingers, leaving the fifth dry. Place crushed ice around the impingers. Add more ice during the run to keep the temperature of the gases leaving the last impinger at about 20° C (70° F), or less.

4.1.2 Purge the connecting line between the sampling valve and the first impinger. Connect the sample line to the train. Record the initial reading on the dry gas meter as shown in Table 11-1.

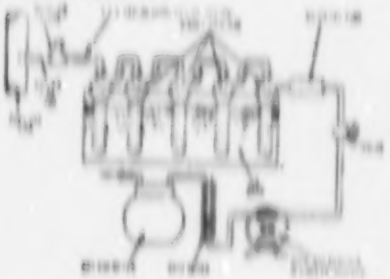

Figure 11-1

Table 11-1.—Field data

Location ..............
Test ..............
Date ..............
Operator ..............
Barometric pressure ..............
Comments:

| Clock time | Gas volume through meter (Vm), liters (cubic feet) | Rotameter setting, liters per minute (cubic feet per minute) | Meter temperature, °C (°F) |
|---|---|---|---|
| | | | |

4.1.3 Open the flow control valve and adjust the sampling rate to 1.13 liters per minute (0.04 cfm). Read the meter temperature and record on Table 11-1.

4.1.4 Continue sampling a minimum of 10 minutes. If the yellow color of cadmium sulfide is visible in the third impinger, analysis should confirm that the applicable standard has been exceeded. At the end of the sample time, close the flow control valve and read the final meter volume and temperature.

4.1.5 Disconnect the impinger train from the sampling line. Purge the train with clean ambient air for 15 minutes to ensure that all $H_2S$ is removed from the hydrogen peroxide. Cap the open ends and move to the sample clean-up area.

4.2 Sample recovery.

4.2.1 Pipette 50 ml of 0.01 N iodine solution into a 250 ml beaker. Add 10 ml of 10 percent HCl to the solution. Mix well.

4.2.2 Discard the contents of the hydrogen peroxide impinger. Carefully transfer the contents of the remaining four impingers to a 500 ml iodine flask.

4.2.3 Rinse the four absorbing impingers and connecting glassware with three portions of the acidified iodine solution. Use the entire 100 ml of acidified iodine for this purpose. Immediately after pouring the acidified iodine into an impinger, stopper it and shake for a few moments before transferring the rinse to the iodine flask. Do not transfer any rinse portion from one impinger to another; transfer it directly to the iodine flask. Once acidified iodine solution has been poured into any glassware containing cadmium sulfide sample, the container must be tightly stoppered at all times except when adding more solution, and this must be done as quickly and carefully as possible. After adding any acidified iodine solution to the iodine flask, allow a few minutes for absorption of the $H_2S$ into the iodine before adding any further rinses.

4.2.4 Follow this rinse with two more rinses using distilled water. Add the distilled water rinses to the iodine flask. Stopper the flask and shake well. Allow about 30 minutes for absorption of the $H_2S$ into the iodine, then complete the analysis titration.

*Caution:* Keep the iodine flask stoppered except when adding sample or titrant.

4.2.5 Prepare a blank in an iodine flask using 45 ml of the absorbing solution, 50 ml of 0.01 N iodine solution, and 10 ml of 10 percent HCl. Stopper the flask, shake well and analyze with the samples.

4.3 Analysis.

*Note:* This analysis titration should be conducted at the sampling location in order to prevent loss of iodine from the sample. Titration should never be made in direct sunlight.

4.3.1 Titrate the solution in the flask with 0.01 N sodium thiosulfate solution until the solution is light yellow. Add 4 ml of the starch indicator solution and continue titrating until the blue color just disappears.

4.3.2 Titrate the blanks in the same manner as the samples.

5. *Calculations.*

5.1 *Normality of the standard iodine solution.*

$$N_I=\frac{N_T V_T}{V_I} \qquad \text{equation 11-1}$$

where:

$N_I$ = normality of iodine, g-eq/liter.
$V_I$ = volume of iodine used, ml.
$N_T$ = normality of sodium thiosulfate, g-eq/liter.
$V_T$ = volume of sodium thiosulfate used, ml.

5.2 *Normality of the standard thiosulfate solution.*

$$N_T = 2.04 \frac{W}{V_T} \qquad \text{equation 11-2}$$

where:

$W$ = weight of $K_2Cr_2O_7$ used, g.
$V_T$ = volume of $Na_2S_2O_3$ used, ml.
$N_T$ = normality of standard thiosulfate solution, g-eq/liter.
2.04 = conversion factor

$$= \frac{(6 \text{ eq } I_2/\text{mole } K_2Cr_2O_7)\ (1{,}000 \text{ ml/l})}{(294.2 \text{ g } K_2Cr_2O_7/\text{mole})\ (10 \text{ aliquot factor})}$$

5.3 *Dry gas volume.* Correct the sample volume measured by the dry gas meter to standard conditions (21° C (70° F)) and 760 mm (29.92 in.) Hg) by using equation 11-3.

$$V_{m_{(std)}} = V_m \left(\frac{T_{std}}{T_m}\right)\left(\frac{P_{bar}}{P_{std}}\right) \qquad \text{equation 11-3}$$

where:

$V_{m_{(std)}}$ = volume at standard conditions of gas sample through the dry gas meter, standard liters (scf).
$V_m$ = volume of gas sample through the dry gas meter (meter conditions), liters (cu. ft.).
$T_{std}$ = absolute temperature at standard conditions, 294° K (530° R).
$T_m$ = average dry gas meter temperature, °K (°R).
$P_{bar}$ = barometric pressure at the orifice meter, mm Hg (in. Hg).
$P_{std}$ = absolute pressure at standard conditions, 760 mm Hg (29.92 in. Hg).

5.4 *Concentration of $H_2S$.*—Calculate the concentration of $H_2S$ in the gas stream at standard conditions using equation 11-4:

$$C_{H_2S} = \frac{K[(V_I N_I - V_T N_T)_{sample} - (V_I N_I - V_T N_T)_{blank}]}{V_{m_{(std)}}}$$

where (metric units):

$C_{H_2S}$ = concentration of $H_2S$ at standard conditions, mg/dscm.
$K$ = conversion factor = $17.0 \times 10^3$

$$= \frac{(34.07 \text{ g/mole } H_2S)(1{,}000 \text{ l/m}^3)(1{,}000 \text{ mg/g})}{(1{,}000 \text{ ml/l})(2H_2S \text{ eq/mole})}$$

$V_I$ = volume of standard iodine solution, ml.
$N_I$ = normality of standard iodine solution, g-eq/liter.
$V_T$ = volume of standard sodium thiosulfate solution, ml.
$N_T$ = normality of standard sodium thiosulfate solution, g-eq/liter.
$V_{m_{(std)}}$ = dry gas volume at standard conditions, liters.

where (English units):

$$K = 0.263 = \frac{17.0(15.43 \text{ gr/g})}{(1{,}000 \text{ l/m}^3)}$$

$V_{m_{(std)}}$ = scf.
$C_{H_2S}$ = gr/dscf.

6. *References*

6.1 Determination of Hydrogen Sulfide, Ammoniacal Cadmium Chloride Method, API Method 772-54. In: Manual on Disposal of Refinery Wastes, Vol. V: Sampling and Analysis of Waste Gases and Particulate Matter, American Petroleum Institute, Washington, D.C., 1954.

6.2 Tentative Method for Determination of Hydrogen Sulfide and Mercaptan Sulfur in Natural Gas, Natural Gas Processors Association, Tulsa, Oklahoma, NGPA Publication No. 2265-65, 1965.

# METHOD 13A

# Determination Of Total Fluoride Emissions From Stationary Sources—SPADNS Zirconium Lake Method

[illegible]

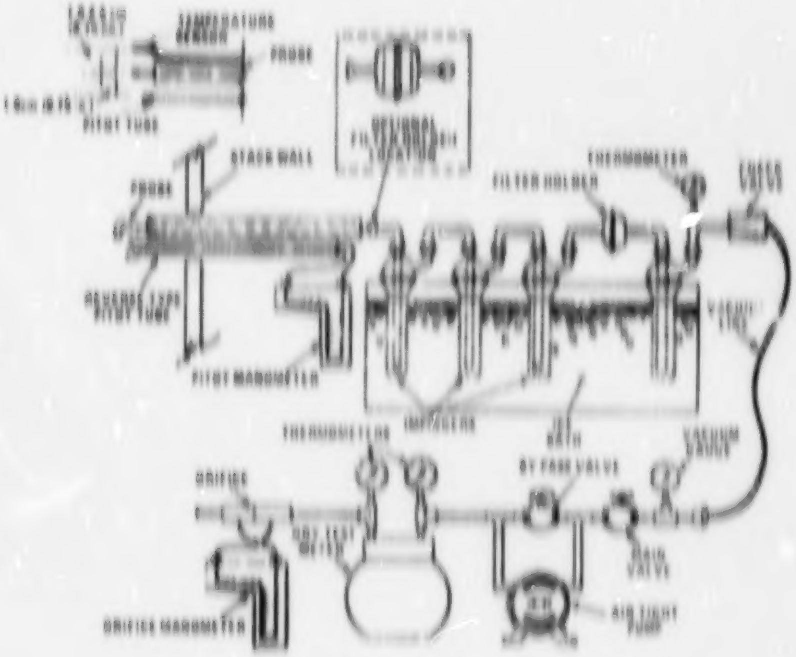
TEMPERATURE SENSOR
PROBE
1.9 cm (0.75 in.)
PITOT TUBE
OPTIONAL FILTER HOLDER LOCATION
STACK WALL
PROBE
REVERSE-TYPE PITOT TUBE
PITOT MANOMETER
FILTER HOLDER
THERMOMETER
CHECK VALVE
VACUUM LINE
IMPINGERS
ICE BATH
THERMOMETERS
BY-PASS VALVE
VACUUM GAUGE
ORIFICE
DRY TEST METER
MAIN VALVE
AIR-TIGHT PUMP
ORIFICE MANOMETER

Figure 13A-1. Fluoride sampling train.

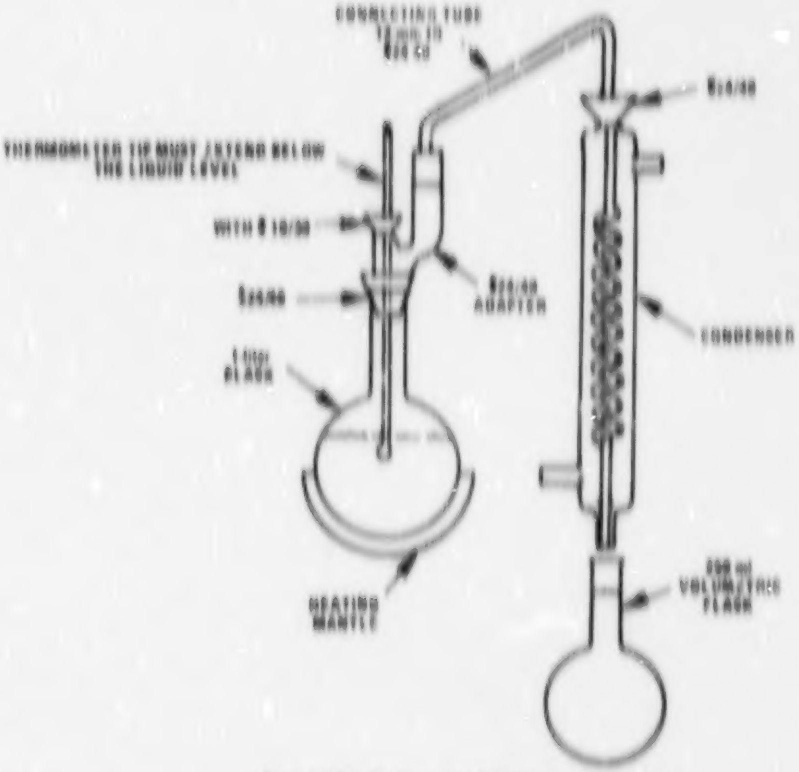
CONNECTING TUBE
12 mm ID
§ 24/40
THERMOMETER TIP MUST EXTEND BELOW THE LIQUID LEVEL
WITH § 10/30
§ 24/40
§ 24/40 ADAPTER
CONDENSER
1 liter FLASK
HEATING MANTLE
250 ml VOLUMETRIC FLASK

Figure 13A-2. Fluoride Distillation Apparatus

2.1.6 Filter heating system—When moisture condensation is a problem, any heating system capable of maintaining a temperature around the filter holder during sampling of 120 ± 14°C (248 ± 25°F). A temperature gauge capable of measuring temperature to within 3°C (5.4°F) shall be installed so that when the filter heater is used, the temperature around the filter holder can be regulated and monitored during sampling. Heating systems other than the one shown in APTD-0581 may be used.

2.1.7 Impingers—Four impingers connected as shown in Figure 13A-1 with ground-glass (or equivalent), vacuum tight fittings. The first, third, and fourth impingers are of the Greenburg-Smith design, modified by replacing the tip with a ½-inch inside diameter glass tube extending to one-half inch from the bottom of the flask. The second impinger is of the Greenburg-Smith design with the standard tip.

2.1.8 Metering system—Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3°C (5.4°F), dry gas meter with 2 percent accuracy at the required sampling rate, and related equipment, or equivalent, as required to maintain an isokinetic sampling rate and to determine sample volume. When the metering system is used in conjunction with a pitot tube, the system shall enable checks of isokinetic rates.

2.1.9 Barometer—Mercury, aneroid, or other barometers capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby weather bureau station, in which case the station value shall be requested and an adjustment for elevation differences shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase.

2.2 Sample recovery.

2.2.1 Probe liner and probe nozzle brushes—Nylon bristles with stainless steel wire handles. The probe brush shall have extensions, at least as long as the probe, of stainless steel, teflon, or similarly inert material. Both brushes shall be properly sized and shaped to brush out the probe liner and nozzle.

2.2.2 Glass wash bottles—Two.

2.2.3 Sample storage containers—Wide mouth, high density polyethylene bottles, 1 liter.

2.2.4 Plastic storage containers—Air tight containers of sufficient volume to store silica gel.

2.2.5 Graduated cylinder—250 ml.

2.2.6 Funnel and rubber policeman—to aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.

2.3 Analysis.

2.3.1 Distillation apparatus—Glass distillation apparatus assembled as shown in Figure 13A-2.

2.3.2 Hot plate—Capable of heating to 500°C.

2.3.3 Electric muffle furnace—Capable of heating to 600°C.

2.3.4 Crucibles—Nickel, 75 to 100 ml capacity.

2.3.5 Beaker, 1500 ml.

2.3.6 Volumetric flask—50 ml.

2.3.7 Erlenmeyer flask or plastic bottle—500 ml.

2.3.8 Constant temperature bath—Capable of maintaining a constant temperature of ±1.0°C in the range of room temperature.

2.3.9 Balance—300 g capacity to measure to ±0.5 g.

2.3.10 Spectrophotometer — Instrument capable of measuring absorbance at 570 nm and providing at least a 1 cm light path.

2.3.11 Spectrophotometer cells—1 cm.

3. Reagents

3.1 Sampling.

3.1.1 Filters—Whatman No. 1 filters, or equivalent, sized to fit filter holder.

3.1.2 Silica gel—Indicating type, 6-16 mesh. If previously used, dry at 175°C (350°F) for 2 hours. New silica gel may be used as received.

3.1.3 Water—Distilled.

3.1.4 Crushed ice.

3.1.5 Stopcock grease—Acetone insoluble, heat stable silicone grease. This is not necessary if screw-on connectors with teflon sleeves, or similar, are used.

3.2 Sample recovery.

3.2.1 Water—Distilled from same container as 3.1.3.

3.3 Analysis.

3.3.1 Calcium oxide (CaO)—Certified grade containing 0.005 percent fluoride or less.

3.3.2 Phenolphthalein Indicator—0.1 percent in 1:1 ethanol-water mixture.

3.3.3 Silver sulfate ($Ag_2SO_4$)—ACS reagent grade, or equivalent.

3.3.4 Sodium hydroxide (NaOH)—Pellets, ACS reagent grade, or equivalent.

3.3.5 Sulfuric acid ($H_2SO_4$)—Concentrated, ACS reagent grade, or equivalent.

3.3.6 Filters—Whatman No. 541, or equivalent.

3.3.7 Hydrochloric acid (HCl)—Concentrated, ACS reagent grade, or equivalent.

3.3.8 Water—Distilled, from same container as 3.1.3.

3.3.9 Sodium fluoride—Standard solution. Dissolve 0.2210 g of sodium fluoride in 1 liter of distilled water. Dilute 100 ml of this solution to 1 liter with distilled water. One milliliter of the solution contains 0.01 mg of fluoride.

3.3.10 SPADNS solution—[4,5 dihydroxy-3-(p-sulfophenylazo)-2,7-naphthalene-disulfonic acid trisodium salt]. Dissolve 0.960 ± .010 g of SPADNS reagent in 500 ml distilled water. This solution is stable for at least one month, if stored in a well-sealed bottle protected from sunlight.

3.3.11 Reference solution—Add 10 ml of SPADNS solution (3.3.10) to 100 ml distilled water and acidify with a solution prepared by diluting 7 ml of concentrated HCl to 10 ml with distilled water. This solution is used to set the spectrophotometer zero point and should be prepared daily.

3.3.12 SPADNS Mixed Reagent—Dissolve 0.135 ± 0.005 g of zirconyl chloride octahydrate ($ZrOCl_2 \cdot 8H_2O$), in 25 ml distilled water. Add 350 ml of concentrated HCl and dilute to 500 ml with distilled water. Mix equal volumes of this solution and SPADNS solution to form a single reagent. This reagent is stable for at least two months.

4. Procedure

Note: The fusion and distillation steps of this procedure will not be required, if it can be shown to the satisfaction of the Administrator that the samples contain only water-soluble fluorides.

4.1 Sampling. The sampling shall be conducted by competent personnel experienced with this test procedure.

4.1.1 Pretest preparation. All train components shall be maintained and calibrated according to the procedure described in APTD-0576, unless otherwise specified herein.

Weigh approximately 200-300 g of silica gel in air tight containers to the nearest 0.5 g. Record the total weight, both silica gel and container, on the container. More silica gel may be used but care should be taken during sampling that it is not entrained and carried out from the impinger. As an alternative, the silica gel may be weighed directly in the impinger or its sampling holder just prior to the train assembly.

4.1.2 Preliminary determinations. Select the sampling site and the minimum number of sampling points according to Method 1 or as specified by the Administrator. Determine the stack pressure, temperature, and the range of velocity heads using Method 2 and moisture content using Approximation Method 4 or its alternatives for the purpose of making isokinetic sampling rate calculations. Estimates may be used. However, final results will be based on actual measurements made during the test.

Select a nozzle size based on the range of velocity heads such that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle size. Ensure that the differential pressure gauge is capable of measuring the minimum velocity head value to within 10%, or as specified by the Administrator.

Select a suitable probe liner and probe length such that all traverse points can be sampled. Consider sampling from opposite sides for large stacks to reduce the length of probes.

Select a total sampling time greater than or equal to the minimum total sampling time specified in the test procedures for the specific industry such that the sampling time per point is not less than 2 min. or select some greater time interval as specified by the Administrator, and such that the sample volume that will be taken will exceed the required minimum total gas sample volume specified in the test procedures for the specific industry. The latter is based on an approximate average sampling rate. Note also that the minimum total sample volume is corrected to standard conditions.

It is recommended that a half-integral or integral number of minutes be sampled at each point in order to avoid timekeeping errors.

In some circumstances, e.g. batch cycles, it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas sample volumes. In these cases, the Administrator's approval must first be obtained.

4.1.3 Preparation of collection train. During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just prior to assembly or until sampling is about to begin.

Place 100 ml of water in each of the first two impingers, leave the third impinger empty, and place approximately 200 g or more, if necessary, of preweighed silica gel in the fourth impinger. Record the weight of the silica gel and container on the data sheet. Place the empty container in a clean place for later use in the sample recovery.

Place a filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as to not allow the sample gas stream to circumvent the filter. Check filter for tears after assembly is completed.

When glass liners are used, install selected nozzle using a Viton A O-ring; the Viton A O-ring is installed as a seal where the nozzle is connected to a glass liner. See APTD-0576 for details. When metal liners are used, install the nozzle as above or by a leak free direct mechanical connection. Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

Unless otherwise specified by the Administrator, attach a temperature probe to the metal sheath of the sampling probe so that the sensor extends beyond the probe tip and does not touch any metal. Its position should be about 1.9 to 2.54 cm (0.75 to 1 in.) from the pitot tube and probe nozzle to avoid interference with the gas flow.

Assemble the train as shown in Figure 13A-1 with the filter between the third and fourth impingers. Alternatively, the filter may be placed between the probe and the first impinger. A filter heating system may be used to prevent moisture condensation, but the temperature around the filter holder shall not exceed 120±14° C (248±25° F). [Note: Whatman No. 1 filter decomposes at 150° C (300° F).] Record filter location on the data sheet.

Place crushed ice around the impingers.

4.1.4 Leak check procedure—After the sampling train has been assembled, turn on and set (if applicable) the probe and filter heating system(s) to reach a temperature sufficient to avoid condensation in the probe. Allow time for the temperature to stabilize. Leak check the train at the sampling site by plugging the nozzle and pulling a 380 mm Hg (15 in. Hg) vacuum. A leakage rate in excess of 4% of the average sampling rate or 0.00057 $m^3$/min. (0.02 cfm), whichever is less, is unacceptable.

The following leak check instructions for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with by-pass valve fully open and coarse adjust valve completely closed. Partially open the coarse adjust valve and slowly close the by-pass valve until 380 mm Hg (15 in. Hg) vacuum is reached. Do not reverse direction of by-pass valve. This will cause water to back up into the filter holder. If 380 mm Hg (15 in. Hg) is exceeded, either leak check at this higher vacuum or end the leak check as described below and start over.

When the leak check is completed, first slowly remove the plug from the inlet to the probe or filter holder and immediately turn off the vacuum pump. This prevents the water in the impingers from being forced backward into the filter holder (if placed before the impingers) and silica gel from being entrained backward into the third impinger.

Leak checks shall be conducted as described whenever the train is disassembled, e.g., for silica gel or filter changes during the test, prior to each test run, and at the completion of each test run. If leaks are found to be in excess of the acceptable rate, the test will be considered invalid. To reduce lost time due to leakage occurrences, it is recommended that leak checks be conducted between port changes.

4.1.5 Particulate train operation—During the sampling run, an isokinetic sampling rate within 10%, or as specified by the Administrator, of true isokinetic shall be maintained.

For each run, record the data required on the example data sheet shown in Figure 13A-2. Be sure to record the initial dry gas meter reading. Record the dry gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made, and when sampling is halted. Take other data point readings at least once at each sample point during each time increment and additional readings when significant changes (20% variation in velocity head readings) necessitate additional adjustments in flow rate. Be sure to level and zero the manometer.

Clean the portholes prior to the test run to minimize chance of sampling deposited material. To begin sampling, remove the nozzle cap, verify (if applicable) that the probe heater is working and filter heater is up to temperature, and that the pitot tube and probe are properly positioned. Position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Nomographs are available for sampling trains using type S pitot tubes with 0.85±0.02 coefficients ($C_p$),

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and when sampling in air or a stack gas with equivalent density (molecular weight, $M_d$, equal to 29±4), which aid in the rapid adjustment of the isokinetic sampling rate without excessive computations. APTD-0576 details the procedure for using these nomographs. If $C_p$ and $M_d$ are outside the above stated ranges, do not use the nomograph unless appropriate steps are taken to compensate for the deviations.

When the stack is under significant negative pressure (height of impinger stem), take care to close the coarse adjust valve before inserting the probe into the stack to avoid water backing into the filter holder. If necessary, the pump may be turned on with the coarse adjust valve closed.

When the probe is in position, block off the openings around the probe and porthole to prevent unrepresentative dilution of the gas stream.

Traverse the stack cross section, as required by Method 1 or as specified by the Administrator, being careful not to bump the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe through the portholes to minimize chance of extracting deposited material.

During the test run, make periodic adjustments to keep the probe and (if applicable) filter temperatures at their proper values. Add more ice and, if necessary, salt to the ice bath, to maintain a temperature of less than 20° C (68° F) at the impinger/silica gel outlet, to avoid excessive moisture losses. Also, periodically check the level and zero of the manometer.

If the pressure drop across the filter becomes high enough to make isokinetic sampling difficult to maintain, the filter may be replaced in the midst of a sample run. It is recommended that another complete filter assembly be used rather than attempting to change the filter itself. After the new filter or filter assembly is installed, conduct a leak check. The final emission results shall be based on the summation of all filter catches.

A single train shall be used for the entire sample run, except for filter and silica gel changes. However, if approved by the Administrator, two or more trains may be used for a single test run when there are two or more ducts or sampling ports. The final emission results shall be based on the total of all sampling train catches.

At the end of the sample run, turn off the pump, remove the probe and nozzle from the stack, and record the final dry gas meter reading. Perform a leak check.[1] Calculate percent isokinetic (see calculation section) to determine whether another test run should be made. If there is difficulty in maintaining isokinetic rates due to source conditions, consult with the Administrator for possible variance on the isokinetic rates.

7.3 Sample recovery. Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period.

When the probe can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over it to keep from losing part of the sample. Do not cap off the probe tip tightly while the sampling train is cooling down, as this would create a vacuum in the filter holder, thus drawing water from the impingers into the filter.

Before moving the sample train to the cleanup site, remove the probe from the sample train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate, if present. Wipe off the silicone grease from the filter inlet where the probe was fastened and cap it. Remove the umbilical cord from the last impinger and cap the impinger. After wiping off the silicone grease, cap off the filter holder outlet and impinger inlet. Ground glass stoppers, plastic caps, or serum caps may be used to close these openings.

Transfer the probe and filter-impinger assembly to the cleanup area. This area should be clean and protected from the wind so that the chances of contaminating or losing the sample will be minimized.

Inspect the train prior to and during disassembly and note any abnormal conditions. Using a graduated cylinder, measure and record the volume of the water in the first three impingers, to the nearest ml; any condensate in the probe should be included in this determination. Treat the samples as follows:

7.3.1 Container No. 1. Transfer the impinger water from the graduated cylinder to this container. Add the filter to this container. Wash all sample exposed surfaces, including the probe tip, probe, first three impingers, impinger connectors, filter holder, and graduated cylinder thoroughly with distilled water. Wash each component three separate times with water and clean the probe and nozzle with brushes. A maximum wash of 500 ml is used, and the washings are added to the sample container which must be made of polyethylene.

7.3.2 Container No. 2. Transfer the silica gel from the fourth impinger to this container and seal.

7.4 Analysis. Treat the contents of each sample container as described below.

7.4.1 Container No. 1.

7.4.1.1 Filter this container's contents, including the Whatman No. 1 filter, through Whatman No. 541 filter paper, or equivalent into a 1500 ml beaker. Note: If filtrate volume exceeds 900 ml make filtrate basic with NaOH to phenolphthalein and evaporate to less than 900 ml.

7.4.1.2 Place the Whatman No. 541 filter containing the insoluble matter (including the Whatman No. 1 filter) in a nickel crucible, add a few ml of water and macerate the filter with a glass rod.

Add 100 mg CaO to the crucible and mix the contents thoroughly to form a slurry. Add a couple of drops of phenolphthalein indicator. The indicator will turn red in a basic medium. The slurry should remain basic during the evaporation of the water or fluoride ion will be lost. If the indicator turns colorless during the evaporation, an acidic condition is indicated. If this happens add CaO until the color turns red again.

Place the crucible in a hood under infrared lamps or on a hot plate at low heat. Evaporate the water completely.

After evaporation of the water, place the crucible on a hot plate under a hood and slowly increase the temperature until the paper chars. It may take several hours for complete charring of the filter to occur.

Place the crucible in a cold muffle furnace and gradually (to prevent smoking) increase the temperature to 600° C, and maintain until the contents are reduced to an ash. Remove the crucible from the furnace and allow it to cool.

7.4.1.3 Add approximately 4 g of crushed NaOH to the crucible and mix. Return the crucible to the muffle furnace, and fuse the sample for 10 minutes at 600° C.

Remove the sample from the furnace and cool to ambient temperature. Using several rinsings of warm distilled water transfer the contents of the crucible to the beaker containing the filtrate from container No. 1 (7.4.1.1). To assure complete sample removal, times thoroughly with two 50 ml portions of 50 percent (V/V) sulfuric acid and carefully add to the beaker. Mix well and transfer to a one-liter volumetric flask. Dilute to volume with distilled water and mix thoroughly. Allow any undissolved solids to settle.

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[1] With acceptability of the test run to be based on the same criterion as in 7.1.4.

7.3.4 Container No. 3. Weigh the spent silica gel and report to the nearest 0.5 g.

7.3.5 Adjustment of acid/water ratio in distillation flask—(Utilize a protective shield when carrying out this procedure.) Place 400 ml of distilled water in the distilling flask and add 200 ml of concentrated $H_2SO_4$. Caution: Observe standard precautions when mixing the $H_2SO_4$ by slowly adding the acid to the flask with constant swirling. Add some soft glass beads and several small pieces of broken glass tubing and assemble the apparatus as shown in Figure 13A-2. Heat the flask until it reaches a temperature of 175 °C to adjust the acid/water ratio for subsequent distillations. Discard the distillate.

7.3.6 Distillation—Cool the contents of the distillation flask to below 80 °C. Pipette an aliquot of sample containing less than 0.6 mg F directly into the distilling flask and add distilled water to make a total volume of 220 ml added to the distilling flask. [For an estimate of what size aliquot does not exceed 0.6 mg F, select an aliquot of the solution and treat as described in Section 7.3.7. This will give an approximation of the fluoride content, but only an approximation since interfering ions have not been removed by the distillation step.]

Place a 250 ml volumetric flask at the condenser exit. Now begin distillation and gradually increase the heat and collect all the distillate up to 175 °C. Caution: Heating the solution above 175 °C will cause sulfuric acid to distill over.

The acid in the distilling flask can be used until there is carryover of interferences or poor fluoride recovery. An occasional check of fluoride recovery with standard solutions is advised. The acid should be changed whenever there is less than 90 percent recovery or blank values are higher than 0.1 μg/ml. Note: If the sample contains chloride, add 5 mg $Ag_2SO_4$ to the flask for every mg of chloride. Gradually increase the heat and collect all the distillate up to 175 °C. Do not exceed 175 °C.

7.3.7 Determination of Concentration—Bring the distillate in the 250 ml volumetric flask to the mark with distilled water and mix thoroughly. Pipette a suitable aliquot from the distillate (containing 10 μg to 40 μg fluoride) and dilute to 50 ml with distilled water. Add 10 ml of SPADNS Mixed Reagent (see Section 6.3.10) and mix thoroughly.

After mixing, place the sample in a constant temperature bath containing the standard solution for thirty minutes before reading the absorbance with the spectrophotometer.

Set the spectrophotometer to zero absorbance at 570 nm with reference solution (6.3.11), and check the spectrophotometer calibration with the standard solution. Determine the absorbance of the samples and determine the concentration from the calibration curve. If the concentration does not fall within the range of the calibration curve, repeat the procedure using a different size aliquot.

8. Calibration

Maintain a laboratory log of all calibrations.

8.1 Sampling Train.

8.1.1 Probe nozzle—Using a micrometer, measure the inside diameter of the nozzle to the nearest 0.025 mm (0.001 in.). Make 3 separate measurements using different diameters each time and obtain the average of the measurements. The difference between the high and low numbers shall not exceed 0.1 mm (0.004 in.).

When nozzles become nicked, dented, or corroded, they shall be reshaped, sharpened, and recalibrated before use.

Each nozzle shall be permanently and uniquely identified.

8.1.2 Pitot tube—The pitot tube shall be calibrated according to the procedure outlined in Method 2.

8.1.3 Dry gas meter and orifice meter. Both meters shall be calibrated according to the procedure outlined in APTD-0576. When diaphragm pumps with by-pass valves are used, check for proper metering system design by calibrating the dry gas meter at an additional flow rate of 0.0057 m³/min. (0.2 cfm) with the by-pass valve fully opened and then with it fully closed. If there is more than ±2 percent difference in flow rates when compared to the fully closed position of the by-pass valve, the system is not designed properly and must be corrected.

8.1.4 Probe heater calibration—The probe heating system shall be calibrated according to the procedure contained in APTD-0576. Probes constructed according to APTD-0581 need not be calibrated if the calibration curves in APTD-0576 are used.

8.1.5 Temperature gauges—Calibrate dial and liquid filled bulb thermometers against mercury-in-glass thermometers. Thermocouples need not be calibrated. For other devices, check with the Administrator.

8.2 Analytical Apparatus. Spectrophotometer. Prepare the blank standard by adding 10 ml of SPADNS mixed reagent to 50 ml of distilled water. Accurately prepare a series of standards from the standard fluoride solution (see Section 6.3.5) by diluting 2, 4, 6, 8, 10, 12 and 14 ml volumes to 100 ml with distilled water. Pipette 50 ml from each solution and transfer to a 100 ml beaker. Then add 10 ml of SPADNS mixed reagent to each. These standards will contain 0, 10, 20, 30, 40, 50, 60, and 70 μg of fluoride (0—1.4 μg/ml) respectively.

After mixing, place the reference standards and reference solution in a constant temperature bath for thirty minutes before reading the absorbance with the spectrophotometer. All samples should be adjusted to this same temperature before analyzing. Since a 3 °C temperature difference between samples and standards will produce an error of approximately 0.005 mg F/liter, care must be taken to see that samples and standards are at nearly identical temperatures when absorbances are recorded.

With the spectrophotometer at 570 nm, use the reference solution (see section 6.3.11) to set the absorbance to zero.

Determine the absorbance of the standards. Prepare a calibration curve by plotting μg F/50 ml versus absorbance on linear graph paper. A standard curve should be prepared initially and thereafter whenever the SPADNS mixed reagent is newly made. Also, a calibration standard should be run with each set of samples and if it differs from the calibration curve by ±2 percent, a new standard curve should be prepared.

9. Calculations

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

9.1 Nomenclature.

$A_d$ = Aliquot of distillate taken for color development, ml.

$A_n$ = Cross sectional area of nozzle, m² (ft²).

$A_t$ = Aliquot of total sample added to still, ml.

$B_{ws}$ = Water vapor in the gas stream, proportion by volume.

$C_s$ = Concentration of fluoride in stack gas, mg/m³, corrected to standard conditions of 20° C, 760 mm Hg (68° F, 29.92 in. Hg) on dry basis.
$F_t$ = Total weight of fluoride in sample, mg.
μg F = Concentration from the calibration curve, μg.
I = Percent of isokinetic sampling.
$m_n$ = Total amount of particulate matter collected, mg.
$M_w$ = Molecular weight of water, 18 g/g-mole (18 lb/lb-mole).
$m_a$ = Mass of residue of acetone after evaporation, mg.
$P_{bar}$ = Barometric pressure at the sampling site, mm Hg (in. Hg).
$P_s$ = Absolute stack gas pressure, mm Hg (in. Hg).
$P_{std}$ = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).
R = Ideal gas constant, 0.06236 mm Hg-m³/°K-g-mole (21.83 in. Hg-ft³/°R-lb-mole).
$T_m$ = Absolute average dry gas meter temperature (see fig. 13A-3), °K (°R).
$T_s$ = Absolute average stack gas temperature (see fig. 13A-3), °K (°R).
$T_{std}$ = Standard absolute temperature, 293° K (528° R).
$V_a$ = Volume of acetone blank, ml.
$V_{aw}$ = Volume of acetone used in wash, ml.
$V_d$ = Volume of distillate collected, ml.
$V_{lc}$ = Total volume of liquid collected in impingers and silica gel, ml. Volume of water in silica gel equals silica gel weight increase in grams times 1 ml/gram. Volume of liquid collected in impinger equals final volume minus initial volume.
$V_m$ = Volume of gas sample as measured by dry gas meter, dcm (dcf).
$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter corrected to standard conditions, dscm (dscf).
$V_{w(std)}$ = Volume of water vapor in the gas sample corrected to standard conditions, scm (scf).
$V_t$ = Total volume of sample, ml.
$v_s$ = Stack gas velocity, calculated by Method 2, Equation 2-7 using data obtained from Method 5, m/sec (ft/sec).
$W_a$ = Weight of residue in acetone wash, mg.
ΔH = Average pressure differential across the orifice (see fig. 13A-3), mm H₂O (in. H₂O).
$\rho_a$ = Density of acetone, mg/ml (see label on bottle).
$\rho_w$ = Density of water, 1 g/ml (0.00220 lb/ml).
θ = Total sampling time, min.
13.6 = Specific gravity of mercury.
60 = Sec/min.
100 = Conversion to percent.

**9.2 Average dry gas meter temperature and average orifice pressure drop.** See data sheet (fig. 13A-3).

**9.3 Dry gas volume.** Correct the sample volume measured by the dry gas meter to standard conditions (20° C, 760 mm Hg (68° F, 29.92 inches Hg)) by using equation 13A-1.

$$V_{m(std)}=V_m\frac{T_{std}}{T_m}\left[\frac{P_{bar}+\frac{\Delta H}{13.6}}{P_{std}}\right]=KV_m\frac{P_{bar}+\Delta H/13.6}{T_m}$$

equation 13A-1

where:
K = 0.3855 °K/mm Hg for metric units.
= 17.65 °R/in. Hg for English units.

**9.4 Volume of water vapor.**

$$V_{w(std)}=V_{lc}\frac{\rho_w}{M_w}\frac{RT_{std}}{P_{std}}=KV_{lc}$$ equation 13A-2

where:
K = 0.00134 m³/ml for metric units.
= 0.0472 ft³/ml for English units.

**9.5 Moisture content.**

$$B_{ws}=\frac{V_{w(std)}}{V_{m(std)}+V_{w(std)}}$$

equation 13A-3

If the liquid droplets are present in the gas stream assume the stream to be saturated and use a psychrometric chart to obtain an approximation of the moisture percentage.

9.6 Concentration.

9.6.1 Calculate the amount of fluoride in the sample according to Equation 13A-4.

$$F_t=K\frac{V_t}{A_t}\frac{V_d}{A_d}(\mu g\ F)$$

equation 13A-4

where:
K = 10⁻³ mg/μg

9.6.2 Concentration of fluoride in stack gas. Determine the concentration of fluoride in the stack gas according to Equation 13A-5.

$$C_s=K\frac{F_t}{V_{m(std)}}$$

equation 13A-5

where:
K = 35.31 ft³/m³.

6.7 Isokinetic variation.

6.7.1 Calculations from raw data.

$$I = \frac{100\,T_s\,[K V_{1c} + (V_m/T_m)\,(P_{bar} + \Delta H/13.6)]}{60\,\theta\,v_s\,P_s\,A_n} \qquad \text{equation 13A-6}$$

where:

K = 0.00346 mm Hg-m³/ml-°K for metric units

= 0.00267 in. Hg-ft³/ml-°R for English units

6.7.2 Calculations from intermediate values.

$$I = \frac{T_s\,V_{m(std)}\,P_{std}\,100}{T_{std}\,v_s\,\theta\,A_n\,P_s\,60\,(1-B_{ws})}$$

$$= K\,\frac{T_s\,V_{m(std)}}{P_s\,v_s\,A_n\,\theta\,(1-B_{ws})} \qquad \text{equation 13A-7}$$

where:

K = 4.320 for metric units.

= 0.09450 for English units.

6.8 *Acceptable results.* The following range sets the limit on acceptable isokinetic sampling results:

If 90 percent ≤ I ≤ 110 percent, the results are acceptable. If the results are low in comparison to the standards and I is beyond the acceptable range, the Administrator may option to accept the results. Use reference 7.4 to make judgments. Otherwise, reject the results and repeat the test.

7. *References.*

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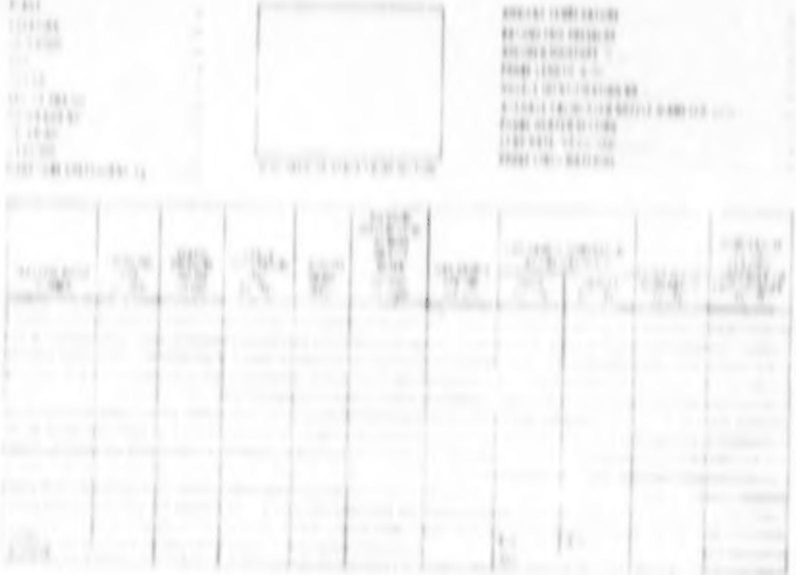

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Reference Method 13A is amended as follows:

(a) In section 3, the phrase "200 µg/liter" is corrected to read "200 mg/liter" and the parenthetical phrase "(see section 7.2.2)" is corrected to read "(see section 7.2.4)".

(b) Section 5.1.5 is revised to read as follows:

5.1.5 Filter holder—If located between the probe and first impinger, borosilicate glass with a 20 mesh stainless steel screen filter support and a silicone rubber gasket; neither a glass frit filter support nor a sintered metal filter support may be used if the filter is in front of the impingers. If located between the third and fourth impingers, borosilicate glass with a glass frit filter support and a silicone rubber gasket. Other materials of construction may be used with approval from the Administrator, e.g., if probe liner is stainless steel, then filter holder may be stainless steel. The holder design shall provide a positive seal against leakage from the outside or around the filter.

(c) Section 7.1.5 is amended by revising the first two sentences of the sixth paragraph to read as follows:

7.1.5 Preparation of collection train. * * *

Assemble the train as shown in Figure 13A-1 with the filter between the third and fourth impingers. Alternatively, the filter may be placed between the probe and first impinger if a 20 mesh stainless steel screen is used for the filter support. * * *

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(d) In section 7.3.4, the reference in the first paragraph to "section 7.2.3" is corrected to read "section 7.2.2".

# METHOD 13B

## Determination Of Total Fluoride Emissions From Stationary Sources—Specific Ion Electrode Method

1. *Principle and Applicability.*

1.1 Principle. Gaseous and particulate fluorides are withdrawn isokinetically from the source using a sampling train. The fluorides are collected in the impinger water and on the filter of the sampling train. The weight of total fluorides in the train is determined by the specific ion electrode method.

1.2 Applicability. This method is applicable for the determination of fluoride emissions from stationary sources only when specified by the test procedures for determining compliance with new source performance standards. Fluorocarbons such as Freons, are not quantitatively collected or measured by this procedure.

2. *Range and Sensitivity.*

The fluoride specific ion electrode analytical method covers the range of 0.02-2,000 μg F/ml; however, measurements of less than 0.1 μg F/ml require extra care. Sensitivity has not been determined.

3. *Interferences.*

During the laboratory analysis, aluminum in excess of 300 mg/liter and silicon dioxide in excess of 300 μg/liter will prevent complete recovery of fluoride.

4. *Precision, Accuracy and Stability.*

The accuracy of fluoride electrode measurements has been reported by various researchers to be in the range of 1-5 percent in a concentration range of 0.04 to 80 mg/l. A change in the temperature of the sample will change the electrode response; a change of 1°C will produce a 1.5 percent relative error in the measurement. Lack of stability in the electrometer used to measure EMF can introduce error. An error of 1 millivolt in the EMF measurement produces a relative error of 4 percent regardless of the absolute concentration being measured.

5. *Apparatus.*

5.1 Sample train. See Figure 13A-1 (Method 13A); it is similar to the Method 5 train except for the interchangeability of the position of the filter. Commercial models of this train are available. However, if one desires to build his own, complete construction details are described in APTD-0581; for changes from the APTD-0581 document and for allowable modifications to Figure 13A-1, see the following subsections.

The operating and maintenance procedures for the sampling train are described in APTD-0576. Since correct usage is important in obtaining valid results, all users should read the APTD-0576 document and adopt the operating and maintenance procedures outlined in it, unless otherwise specified herein.

5.1.1 Probe nozzle—Stainless steel (316) with sharp, tapered leading edge. The angle of taper shall be ≤30° and the taper shall be on the outside to preserve a constant internal diameter. The probe nozzle shall be of the button-hook or elbow design, unless otherwise specified by the Administrator. The wall thickness of the nozzle shall be less than or equal to that of 20 gauge tubing, i.e., 0.165 cm (0.065 in.) and the distance from the tip of the nozzle to the first bend or point of disturbance shall be at least two times the outside nozzle diameter. The nozzle shall be constructed from seamless stainless steel tubing. Other configurations and construction material may be used with approval from the Administrator.

A range of sizes suitable for isokinetic sampling should be available, e.g., 0.32 cm (1/8 in.) up to 1.27 cm (1/2 in.) (or larger if higher volume sampling trains are used) inside diameter (ID) nozzles in increments of 0.16 cm (1/16 in.). Each nozzle shall be calibrated according to the procedures outlined in the calibration section.

5.1.2 Probe liner—Borosilicate glass or stainless steel (316). When the filter is located immediately after the probe, a probe heating system may be used to prevent filter plugging resulting from moisture condensation. The temperature in the probe shall not exceed 120±14°C (248±25°F).

5.1.3 Pitot tube—Type S, or other device approved by the Administrator, attached to probe to allow constant monitoring of the stack gas velocity. The face openings of the pitot tube and the probe nozzle shall be adjacent and parallel to each other, not necessarily on the same plane, during sampling. The free space between the nozzle and pitot tube shall be at least 1.9 cm (0.75 in.). The free space shall be set based on a 1.3 cm (0.5 in.) ID nozzle, which is the largest size nozzle used.

The pitot tube must also meet the criteria specified in Method 2 and be calibrated according to the procedure in the calibration section of that method.

5.1.4 Differential pressure gauge—Inclined manometer capable of measuring velocity head to within 10 percent of the minimum measured value. Below a differential pressure of 1.3 mm (0.05 in.) water gauge, micromanometers with sensitivities of 0.013 mm (0.0005 in.) should be used. However, micromanometers are not easily adaptable to field conditions and are not easy to use with pulsating flow. Thus, other methods or devices acceptable to the Administrator may be used when conditions warrant.

5.1.5 Filter holder—Borosilicate glass with a glass frit filter support and a silicone rubber gasket. Other materials of construction may be used with approval from the Administrator, e.g., if probe liner is stainless steel, then filter holder may be stainless steel. The holder design shall provide a positive seal against leakage from the outside or around the filter.

5.1.6 Filter heating system—When moisture condensation is a problem, any heating system capable of maintaining a temperature around the filter holder during sampling of no greater than 120±14°C (248±25°F). A temperature gauge capable of measuring temperature to within 3°C (5.4°F) shall be installed so that when the filter heater is used, the temperature around the filter holder can be regulated and monitored during sampling.

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Heating systems other than the one shown in APTD-0581 may be used.

5.1.7 Impingers—Four impingers connected as shown in Figure 13A-1 with ground glass (or equivalent), vacuum tight fittings. The first, third, and fourth impingers are of the Greenburg-Smith design, modified by replacing the tip with a 1¼ cm (½ in.) inside diameter glass tube extending to 1¼ cm (½ in.) from the bottom of the flask. The second impinger is of the Greenburg-Smith design with the standard tip.

5.1.8 Metering system—Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3°C (~5°F), dry gas meter with 2 percent accuracy at the required sampling rate, and related equipment, or equivalent, as required to maintain an isokinetic sampling rate and to determine sample volume. When the metering system is used in conjunction with a pitot tube, the system shall enable checks of isokinetic rates.

5.1.9 Barometer—Mercury, aneroid, or other barometers capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby weather bureau station, in which case the station value shall be requested and an adjustment for elevation differences shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase.

5.2 Sample recovery.

5.2.1 Probe liner and probe nozzle brushes—Nylon bristles with stainless steel wire handles. The probe brush shall have extensions, at least as long as the probe, of stainless steel, teflon, or similarly inert material. Both brushes shall be properly sized and shaped to brush out the probe liner and nozzle.

5.2.2 Glass wash bottles—Two.

5.2.3 Sample storage containers—Wide mouth, high density polyethylene bottles, 1 liter.

5.2.4 Plastic storage containers—Air tight containers of sufficient volume to store silica gel.

5.2.5 Graduated cylinder—250 ml.

5.2.6 Funnel and rubber policeman—to aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.

5.3 Analysis.

5.3.1 Distillation apparatus—Glass distillation apparatus assembled as shown in Figure 13A-2 (Method 13A).

5.3.2 Hot plate—Capable of heating to 500°C.

5.3.3 Electric muffle furnace—Capable of heating to 600°C.

5.3.4 Crucibles—Nickel, 75 to 100 ml capacity.

5.3.5 Beaker—1500 ml.

5.3.6 Volumetric flask—50 ml.

5.3.7 Erlenmeyer flask or plastic bottle—500 ml.

5.3.8 Constant temperature bath—Capable of maintaining a constant temperature of ±1.0°C in the range of room temperature.

5.3.9 Trip balance—300 g capacity to measure to ±0.5 g.

5.3.10 Fluoride ion activity sensing electrode.

5.3.11 Reference electrode—Single junction; sleeve type. (A combination-type electrode having the reference electrode and the fluoride-ion sensing electrode built into one unit may also be used.)

5.3.12 Electrometer—A pH meter with millivolt scale capable of ±0.1 mv resolution, or a specific ion meter made specifically for specific ion use.

5.3.13 Magnetic stirrer and TFE fluorocarbon coated stripping bars.

6. Reagents.

6.1 Sampling.

6.1.1 Filters—Whatman No. 1 filters, or equivalent, sized to fit filter holder.

6.1.2 Silica gel—Indicating type, 6-16 mesh. If previously used, dry at 175°C (350°F) for 2 hours. New silica gel may be used as received.

6.1.3 Water—Distilled.

6.1.4 Crushed ice.

6.1.5 Stopcock grease—Acetone insoluble, heat stable silicone grease. This is not necessary if screw-on connectors with teflon sleeves, or similar, are used.

6.2 Sample recovery.

6.2.1 Water—Distilled from same container as 6.1.3.

6.3 Analysis.

6.3.1 Calcium oxide (CaO)—Certified grade containing 0.005 percent fluoride or less.

6.3.2 Phenolphthalein Indicator—0.1 percent in 1:1 ethanol water mixture.

6.3.3 Sodium hydroxide (NaOH)—Pellets, ACS reagent grade or equivalent.

6.3.4 Sulfuric acid ($H_2SO_4$)—Concentrated, ACS reagent grade or equivalent.

6.3.5 Filters—Whatman No. 541, or equivalent.

6.3.6 Water—Distilled, from same container as 6.1.3.

6.3.7 Total Ionic Strength Adjustment Buffer (TISAB)—Place approximately 500 ml of distilled water in a 1-liter beaker. Add 57 ml glacial acetic acid, 58 g sodium chloride, and 4 g CDTA (Cyclohexylene dinitrilo tetraacetic acid). Stir to dissolve. Place the beaker in a water bath to cool it. Slowly add 5 M NaOH to the solution, measuring the pH continuously with a calibrated pH/reference electrode pair, until the pH is 5.3. Cool to room temperature. Pour into a 1-liter flask and dilute to volume with distilled water. Commercially prepared TISAB buffer may be substituted for the above.

6.3.8 Fluoride Standard Solution—0.1 M fluoride reference solution. Add [illegible] grams of reagent grade sodium fluoride (NaF) to a 1-liter volumetric flask and add enough distilled water to dissolve. Dilute to volume with distilled water.

7. *Procedure.*

NOTE: The fusion and distillation steps of this procedure will not be required, if it can be shown to the satisfaction of the Administrator that the samples contain only water-soluble fluorides.

7.1 Sampling. The sampling shall be conducted by competent personnel experienced with this test procedure.

7.1.1 Pretest preparation. All train components shall be maintained and calibrated according to the procedure described in APTD-0576, unless otherwise specified herein.

Weigh approximately 200-300 g of silica gel in air tight containers to the nearest 0.5 g. Record the total weight, both silica gel and container, on the container. More silica gel may be used but care should be taken during sampling that it is not entrained and carried out from the impinger. As an alternative, the silica gel may be weighed directly in the impinger or its sampling holder just prior to the train assembly.

7.1.2 Preliminary determinations. Select the sampling site and the minimum number of sampling points according to Method 1 or as specified by the Administrator. Determine the stack pressure, temperature, and the range of velocity heads using Method 2 and moisture content using Approximation Method 4 or its alternatives for the purpose of making isokinetic sampling rate calculations. Estimates may be used. However, final results will be based on actual measurements made during the test.

Select a nozzle size based on the range of velocity heads such that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle size. Ensure that the differential pressure gauge is capable of measuring the minimum velocity head value to within 10 percent, or as specified by the Administrator.

Select a suitable probe liner and probe length such that all traverse points can be sampled. Consider sampling from opposite sides for large stacks to reduce the length of probes.

Select a total sampling time greater than or equal to the minimum total sampling time specified in the test procedures for the specific industry such that the sampling time per point is not less than 2 min (or select some greater time interval as specified by the Administrator), and such that the sample volume that will be taken will exceed the required minimum total gas sample volume specified in the test procedures for the specific industry. The latter is based on an approximate average sampling rate. Note also that the minimum total sample volume is corrected to standard conditions.

It is recommended that a half-integral or integral number of minutes be sampled at each point in order to avoid timekeeping errors.

In some circumstances, e.g. batch cycles, it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas sample volumes. In these cases, the Administrator's approval must first be obtained.

7.1.3 Preparation of collection train. During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just prior to assembly or until sampling is about to begin.

Place 100 ml of water in each of the first two impingers, leave the third impinger empty, and place approximately 200 to 300 g or more, if necessary, of preweighed silica gel in the fourth impinger. Record the weight of the silica gel and container on the data sheet. Place the empty container in a clean place for later use in the sample recovery.

Place a filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as to not allow the sample gas stream to circumvent the filter. Check filter for tears after assembly is completed.

When glass liners are used, install selected nozzle using a Viton A O-ring; the Viton A O-ring is installed as a seal where the nozzle is connected to a glass liner. See APTD-0576 for details. When metal liners are used, install the nozzle as above or by a leak free direct mechanical connection. Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

Unless otherwise specified by the Administrator, attach a temperature probe to the metal sheath of the sampling probe so that the sensor extends beyond the probe tip and does not touch any metal. Its position should be about 1.90 to 2.54 cm (0.75 to 1 in.) from the pitot tube and probe nozzle to avoid interference with the gas flow.

Assemble the train as shown in Figure 13A-1 (Method 13A) with the filter between the third and fourth impingers. Alternatively, the filter may be placed between the probe and first impinger. A filter heating system may be used to prevent moisture condensation, but the temperature around the filter holder shall not exceed 120±14° C (248±25° F). (Note: Whatman No. 1 filter decomposes at 150° C (300° F).) Record filter location on the data sheet.

Place crushed ice around the impingers.

7.1.4 Leak check procedure. After the sampling train has been assembled, turn on and set (if applicable) the probe and filter heating system(s) to reach a temperature sufficient to avoid condensation in the probe. Allow time for the temperature to stabilize. Leak check the train at the sampling site by plugging the nozzle and pulling a 380 mm Hg (15 in. Hg) vacuum. A leakage rate in excess of 4% of the average sampling rate or 0.00057 m³/min (0.02 cfm), whichever is less, is unacceptable.

The following leak check instruction for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with by-pass valve fully open and coarse adjust valve completely closed. Partially open the coarse adjust valve and slowly close the by-pass valve until 380 mm Hg (15 in. Hg) vacuum is reached. Do not reverse direction of by-pass valve. This will cause water to back up into the filter holder. If 380 mm Hg (15 in. Hg) is exceeded, either leak check at this higher vacuum or end the leak check as described below and start over.

When the leak check is completed, first slowly remove the plug from the inlet to the probe or filter holder and immediately turn off the vacuum pump. This prevents the water in the impingers from being forced backward into the filter holder (if placed before the impingers) and silica gel from being entrained backward into the third impinger.

Leak checks shall be conducted as described whenever the train is disengaged, e.g., for silica gel or filter changes during the test, prior to each test run, and at the completion of each test run. If leaks are found to be in excess of the acceptable rate, the test will be considered invalid. To reduce lost time due to leakage occurrences, it is recommended that leak checks be conducted between port changes.

7.1.5 Particulate train operation. During the sampling run, an isokinetic sampling rate within 10%, or as specified by the Administrator, of true isokinetic shall be maintained.

For each run, record the data required on the example data sheet shown in Figure 13A-2 (Method 13A). Be sure to record the initial dry gas meter reading. Record the dry gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made, and when sampling is halted. Take other data point readings at least once at each sample point during each time increment and additional readings when significant changes (20% variation in velocity head readings) necessitate additional adjustments in flow rate. Be sure to level and zero the manometer.

Clean the portholes prior to the test run to minimize chance of sampling deposited material. To begin sampling, remove the nozzle cap, verify (if applicable) that the probe heater is working and filter heater is up to temperature, and that the pitot tube and probe are properly positioned. Position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Nomographs are available for sampling trains using type S pitot tubes with 0.85±0.02 coefficients ($C_p$), and when sampling in air or a stack gas with equivalent density (molecular weight, $M_d$, equal to 29±4), which aid in the rapid adjustment of the isokinetic sampling rate without excessive computations. APTD-0576 details the procedure for using these nomographs. If $C_p$ and $M_d$ are outside the above stated ranges, do not use the nomograph unless appropriate steps are taken to compensate for the deviation.

When the stack is under significant negative pressure (height of impinger stem), take care to close the coarse adjust valve before inserting the probe into the stack to avoid water backing into the filter holder. If necessary, the pump may be turned on with the coarse adjust valve closed.

When the probe is in position, block off the openings around the probe and porthole to prevent unrepresentative dilution of the gas stream.

Traverse the stack cross section, as required by Method 1 or as specified by the Administrator, being careful not to bump the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe through the portholes to minimize chance of extracting deposited material.

During the test run, make periodic adjustments to keep the probe and (if applicable) filter temperatures at their proper values. Add more ice and, if necessary, salt to the ice bath, to maintain a temperature of less than 20° C (68° F) at the impinger/silica gel outlet, to avoid excessive moisture losses. Also, periodically check the level and zero of the manometer.

If the pressure drop across the filter becomes high enough to make isokinetic sampling difficult to maintain, the filter may be replaced in the midst of a sample run. It is recommended that another complete filter assembly be used rather than attempting to change the filter itself. After the new filter or filter assembly is installed, conduct a leak check. The final emission results shall be based on the summation of all filter catches.

A single train shall be used for the entire sample run, except for filter and silica gel changes. However, if approved by the Administrator, two or more trains may be used for a single test run when there are two or more ducts or sampling ports. The final emission results shall be based on the total of all sampling train catches.

At the end of the sample run, turn off the pump, remove the probe and nozzle from the stack, and record the final dry gas meter reading. Perform a leak check.[1] Calculate percent isokinetic (see calculation section) to determine whether another test run should be made. If there is difficulty in maintaining isokinetic rates due to source conditions, consult with the Administrator for possible variance on the isokinetic rates.

7.2 Sample recovery. Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period.

When the probe can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over it to keep from losing part of the sample. Do not cap off the probe tip tightly while the sampling train is cooling down, as this would create a vacuum in the filter holder, thus drawing water from the impingers into the filter.

Before moving the sample train to the cleanup site, remove the probe from the sample train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate, if present. Wipe off the silicone grease from the filter inlet where the probe was fastened and cap it. Remove the umbilical cord from the last impinger and cap the impinger. After wiping off the silicone grease, cap off the filter holder outlet and impinger inlet. Ground glass stoppers, plastic caps, or serum caps may be used to close these openings.

Transfer the probe and filter-impinger assembly to the cleanup area. This area should be clean and protected from the wind so that the chances of contaminating or losing the sample will be minimized.

Inspect the train prior to and during disassembly and note any abnormal conditions. Using a graduated cylinder, measure and record the volume of the water in the first three impingers, to the nearest ml; any condensate in the probe should be included in this determination. Treat the samples as follows:

7.2.1 Container No. 1. Transfer the impinger water from the graduated cylinder to this container. Add the filter to this container. Wash all sample exposed surfaces, including the probe tip, probe, first three impingers, impinger connectors, filter holder, and graduated cylinder thoroughly with distilled water. Wash each component three separate times with water and clean the probe and nozzle with brushes. A maximum wash of 500 ml is used, and the washings are added to the sample container which must be made of polyethylene.

7.2.2 Container No. 2. Transfer the silica gel from the fourth impinger to this container and seal.

7.3 Analysis. Treat the contents of each sample container as described below.

7.3.1 Container No. 1.

7.3.1.1 Filter this container's contents, including the Whatman No. 1 filter, through Whatman No. 541 filter paper, or equivalent into a 1500 ml beaker. Note: If filtrate volume exceeds 900 ml make filtrate basic with NaOH to phenolphthalein and evaporate to less than 900 ml.

7.3.1.2 Place the Whatman No. 541 filter containing the insoluble matter (including the Whatman No. 1 filter) in a nickel crucible, add a few ml of water and macerate the filter with a glass rod.

Add 100 mg CaO to the crucible and mix the contents thoroughly to form a slurry. Add a couple of drops of phenolphthalein indicator. The indicator will turn red in a basic medium. The slurry should remain basic during the evaporation of the water or fluoride ion will be lost. If the indicator turns colorless during the evaporation, an acidic condition is indicated. If this happens add CaO until the color turns red again.

Place the crucible in a hood under infrared lamps or on a hot plate at low heat. Evaporate the water completely.

After evaporation of the water, place the crucible on a hot plate under a hood and slowly increase the temperature until the paper chars. It may take several hours for complete charring of the filter to occur.

Place the crucible in a cold muffle furnace and gradually (to prevent smoking) increase the temperature to 600° C, and maintain until the contents are reduced to an ash. Remove the crucible from the furnace and allow it to cool.

7.3.1.3 Add approximately 4 g of crushed NaOH to the crucible and mix. Return the crucible to the muffle furnace, and fuse the sample for 10 minutes at 600° C.

Remove the sample from the furnace and cool to ambient temperature. Using several rinsings of warm distilled water transfer the contents of the crucible to the beaker containing the filtrate from container No. 1 (7.3.1). To assure complete sample removal, rinse finally with two 20 ml portions of 25 percent (v/v) sulfuric acid and carefully add to the beaker. Mix well and transfer to a one-liter volumetric flask. Dilute to volume with distilled water and mix thoroughly. Allow any undissolved solids to settle.

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[1] With acceptability of the test run to be based on the same criterion as in 7.1.4.

7.2.2 Container No. 2. Weigh the spent silica gel and report to the nearest 0.1 g.

7.2.3 Adjustment of acid/water ratio in distillation flask.—Utilize a protective shield when carrying out this procedure. Place 400 ml of distilled water in the distilling flask and add 200 ml of concentrated $H_2SO_4$. Caution: Observe standard precautions when mixing the $H_2SO_4$ by slowly adding the acid to the flask with constant swirling. Add some soft glass beads and several small pieces of broken glass tubing and assemble the apparatus as shown in Figure 13A-2. Heat the flask until it reaches a temperature of 175° C to adjust the acid/water ratio for subsequent distillations. Discard the distillate.

7.2.4 Distillation.—Cool the contents of the distillation flask to below 80° C. Pipette an aliquot of sample containing less than 0.6 mg F directly into the distilling flask and add distilled water to make a total volume of 220 ml added to the distilling flask. [For an estimate of what size aliquot does not exceed 0.6 mg F, select an aliquot of the solution and treat as described in Section 7.2.5. This will give an approximation of the fluoride content, but only an approximation since interfering ions have not been removed by the distillation step.]

Place a 250 ml volumetric flask at the condenser exit. Now begin distillation and gradually increase the heat and collect all the distillate up to 175° C. Caution: Heating the solution above 175° C will cause sulfuric acid to distill over.

The acid in the distilling flask can be used until there is carryover of interferences or poor fluoride recovery. An occasional check of fluoride recovery with standard solutions is advised. The acid should be changed whenever there is less than 90 percent recovery or blank values are higher than 0.1 μg/ml.

7.2.5 Determination of concentration.—Bring the distillate in the 250 ml volumetric flask to the mark with distilled water and mix thoroughly. Pipette a 25 ml aliquot from the distillate. Add an equal volume of TISAB and mix. The sample should be at the same temperature as the calibration standards when measurements are made. If ambient lab temperature fluctuates more than ±2° C from the temperature at which the calibration standards were measured, condition samples and standards in a constant temperature bath before measurement. Stir the sample with a magnetic stirrer during measurement to minimize electrode response time. If the stirrer generates enough heat to change solution temperature, place a piece of insulating material such as cork between the stirrer and the beaker. Dilute samples (below $10^{-4}$ M fluoride ion content) should be held in polyethylene or polypropylene beakers during measurement.

Insert the fluoride and reference electrodes into the solution. When a steady millivolt reading is obtained, record it. This may take several minutes. Determine concentration from the calibration curve. Between electrode measurements, soak the fluoride sensing electrode in distilled water for 30 seconds and then remove and blot dry.

8. Calibration

Maintain a laboratory log of all calibrations.

8.1 Sampling Train

8.1.1 Probe nozzle.—Using a micrometer, measure the inside diameter of the nozzle to the nearest 0.025 mm (0.001 in.). Make 3 separate measurements using different diameters each time and obtain the average of the measurements. The difference between the high and low numbers shall not exceed 0.1 mm (0.004 in.).

When nozzles become nicked, dented, or corroded, they shall be reshaped, sharpened, and recalibrated before use.

Each nozzle shall be permanently and uniquely identified.

8.1.2 Pitot tube.—The pitot tube shall be calibrated according to the procedure outlined in Method 2.

8.1.3 Dry gas meter and orifice meter. Both meters shall be calibrated according to the procedure outlined in APTD-0576. When diaphragm pumps with by-pass valves are used, check for proper metering system design by calibrating the dry gas meter at an additional flow rate of 0.0057 $m^3$/min. (0.2 cfm) with the by-pass valve fully opened and then with it fully closed. If there is more than ±2 percent difference in flow rates when compared to the fully closed position of the by-pass valve, the system is not designed properly and must be corrected.

8.1.4 Probe heater calibration.—The probe heating system shall be calibrated according to the procedure contained in APTD-0576. Probes constructed according to APTD-0581 need not be calibrated if the calibration curves in APTD-0576 are used.

8.1.5 Temperature gauges.—Calibrate dial and liquid filled bulb thermometers against mercury-in-glass thermometers. Thermocouples need not be calibrated. For other devices, check with the Administrator.

8.2 Analytical Apparatus

8.2.1 Fluoride Electrode.—Prepare fluoride standardizing solutions by serial dilution of the 0.1 M fluoride standard solution. Pipette 10 ml of 0.1 M NaF into a 100 ml volumetric flask and make up to the mark with distilled water for a $10^{-2}$ M standard solution. Use 10 ml of $10^{-2}$ M solution to make a $10^{-3}$ M solution in the same manner. Repeat for $10^{-4}$ and $10^{-5}$ M solutions.

Pipette 50 ml of each standard into a separate beaker. Add 50 ml of TISAB to each beaker. Place the electrode in the most dilute standard solution. When a steady millivolt reading is obtained, plot the value on the linear axis of semi-log graph paper versus concentration on the log axis. Plot the nominal value for concentration of the standard on the log axis, e.g., when 50 ml of $10^{-2}$ M standard is diluted with 50 ml TISAB, the concentration is still designated "$10^{-2}$ M".

Between measurements soak the fluoride sensing electrode in distilled water for 30 seconds, and then remove and blot dry. Analyze the standards going from dilute to concentrated standards. A straight-line calibration curve will be obtained, with nominal concentrations of $10^{-5}$, $10^{-4}$, $10^{-3}$, $10^{-2}$, $10^{-1}$ fluoride molarity on the log axis plotted versus electrode potential (in millivolts) on the linear scale.

Calibrate the fluoride electrode daily, and check it hourly. Prepare fresh fluoride standardizing solutions daily of $10^{-2}$ M or less. Store fluoride standardizing solutions in polyethylene or polypropylene containers. (Note: Certain specific ion meters have been designed specifically for fluoride electrode use and give a direct readout of fluoride ion concentration. These meters may be used in lieu of calibration curves for fluoride measurements over narrow concentration ranges. Calibrate the meter according to manufacturer's instructions.)

9. Calculations

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

9.1 Nomenclature.

$A_n$ = Cross sectional area of nozzle, m² (ft²).

$A_t$ = Aliquot of total sample added to still, ml.

$B_{ws}$ = Water vapor in the gas stream, proportion by volume.

$C_s$ = Concentration of fluoride in stack gas, mg/m³, corrected to standard conditions of 20° C, 760 mm Hg (68° F, 29.92 in. Hg) on dry basis.

$F_t$ = Total weight of fluoride in sample, mg.

I = Percent of isokinetic sampling.

M = Concentration of fluoride from calibration curve, molarity.

$m_n$ = Total amount of particulate matter collected, mg.

$M_w$ = Molecular weight of water, 18 g/g-mole (18 lb/lb-mole).

$m_a$ = Mass of residue of acetone after evaporation, mg.

$P_{bar}$ = Barometric pressure at the sampling site, mm Hg (in. Hg).

$P_s$ = Absolute stack gas pressure, mm Hg (in. Hg).

$P_{std}$ = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

R = Ideal gas constant, 0.06236 mm Hg-m³/°K-g-mole (21.85 in. Hg-ft³/°R-lb-mole).

$T_m$ = Absolute average dry gas meter temperature (see fig. 13A-3), °K (°R).

$T_s$ = Absolute average stack gas temperature (see fig. 13A-3), °K (°R).

$T_{std}$ = Standard absolute temperature, 293° K (528° R).

$V_a$ = Volume of acetone blank, ml.

$V_{aw}$ = Volume of acetone used in wash, ml.

$V_d$ = Volume of distillate collected, ml.

$V_{lc}$ = Total volume of liquid collected in impingers and silica gel, ml. Volume of water in silica gel equals silica gel weight increase in grams times 1 ml/gram. Volume of liquid collected in impinger equals final volume minus initial volume.

$V_m$ = Volume of gas sample as measured by dry gas meter, dcm (dcf).

$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter corrected to standard conditions, dscm (dscf).

$V_{w(std)}$ = Volume of water vapor in the gas sample corrected to standard conditions, scm (scf).

$V_t$ = Total volume of sample, ml.

$v_s$ = Stack gas velocity, calculated by Method 2, Equation 2-7 using data obtained from Method 5, m/sec (ft/sec).

$W_a$ = Weight of residue in acetone wash, mg.

$\Delta H$ = Average pressure differential across the orifice (see fig. 13A-3), meter, mm H₂O (in. H₂O).

$\rho_a$ = Density of acetone, mg/ml (see label on bottle).

$\rho_w$ = Density of water, 1 g/ml (0.00220 lb/ml).

$\theta$ = Total sampling time, min.

13.6 = Specific gravity of mercury.

60 = Sec/min.

100 = Conversion to percent.

9.2 Average dry gas meter temperature and average orifice pressure drop. See data sheet (Figure 13A-3 of Method 13A).

9.3 Dry gas volume. Use Section 9.3 of Method 13A.

9.4 Volume of Water Vapor. Use Section 9.4 of Method 13A.

9.5 Moisture Content. Use Section 9.5 of Method 13A.

9.6 Concentration.

9.6.1 Calculate the amount of fluoride in the sample according to equation 13B-1.

$$F_t = K \frac{V_t}{A_t} (V_d)(M)$$

where:

$K$ = 19 mg/ml.

9.6.2 Concentration of fluoride in stack gas. Use Section 9.6.2 of Method 13A.

9.7 Isokinetic variation. Use Section 9.7 of Method 13A.

9.8 Acceptable results. Use Section 9.8 of Method 13A.

## 10. *References*

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**Reference Method 13B is amended as follows:**

**(a) In the third line of section 1, the phrase "300μg/liter" is corrected to read "300 mg/liter".**

**(b) Section 5.1.5 is revised to read as follows:**

5.1.5 Filter holder—If located between the probe and first impinger, borosilicate glass with a 20 mesh stainless steel screen filter support and a silicone rubber gasket; neither a glass frit filter support nor a sintered metal filter support may be used if the filter is in front of the impingers. If located between the third and fourth impingers, borosilicate glass with a glass frit filter support and a silicone rubber gasket. Other materials of construction may be used with approval from the Administrator, e.g., if probe liner is stainless steel, then filter holder may be stainless steel. The holder design shall provide a positive seal against leakage from the outside or around the filter.

**(c) Section 7.1.2 is amended by revising the first two sentences of the sixth paragraph to read as follows:**

7.1.2 Preparation of collection train. * * * Assemble the train as shown in Figure 13A-1 (Method 13A) with the filter between the third and fourth impingers. Alternatively, the filter may be placed between the probe the first impinger if a 20 mesh stainless steel screen is used for the filter support. * * *

* * * * *

**(d) In section 7.3.4, the reference in the first paragraph to "section 7.3.6" is corrected to read "section 7.3.5".**

# METHOD 14

## Determination Of Fluoride Emissions From Potroom Roof Monitors Of Primary Aluminum Plants

1. *Principle and applicability.*

1.1 *Principle.* Gaseous and particulate fluoride roof monitor emissions are drawn into a permanent sampling manifold through several large nozzles. The sample is transported from the sampling manifold to ground level through a duct. The gas in the duct is sampled using Method 13A or 13B—DETERMINATION OF TOTAL FLUORIDE EMISSIONS FROM STATIONARY SOURCES. Effluent velocity and volumetric flow rate are determined with anemometers permanently located in the roof monitor.

1.2 *Applicability.* This method is applicable for the determination of fluoride emissions from stationary sources only when specified by the test procedures for determining compliance with new source performance standards.

2. *Apparatus.*

2.1 *Velocity measurement apparatus.*

2.1.1 *Anemometers.* Vane or propeller anemometers with a velocity measuring threshold as low as 15 meters/minute and a range up to at least 600 meters/minute. Each anemometer shall generate an electrical signal which can be calibrated to the velocity measured by the anemometer. Anemometers shall be able to withstand dusty and corrosive atmospheres.

One anemometer shall be installed for every 85 meters of roof monitor length. If the roof monitor length divided by 85 meters is not a whole number, round the fraction to the nearest whole number to determine the number of anemometers needed. Use one anemometer for any roof monitor less than 85 meters long. Permanently mount the anemometers at the center of each equal length along the roof monitor. One anemometer shall be installed in the same section of the roof monitor that contains the sampling manifold (see section 2.2.1). Make a velocity traverse of the width of the roof monitor where an anemometer is to be placed. This traverse may be made with any suitable low velocity measuring device, and shall be made during normal process operating conditions. Install the anemometer at a point of average velocity along this traverse.

2.1.2 *Recorders.* Recorders equipped with signal transducers for converting the electrical signal from each anemometer to a continuous recording of air flow velocity, or to an integrated measure of volumetric flow. For the purpose of recording velocity, "continuous" shall mean one readout per 15-minute or shorter time interval. A constant amount of time shall elapse between readings. Volumetric flow rate may be determined by an electrical count of anemometer revolutions. The recorders or counters shall permit identification of the velocities or flow rate measured by each individual anemometer.

2.2 *Roof monitor air sampling system.*

2.2.1 *Sampling ductwork.* The manifold system and connecting duct shall be permanently installed to draw an air sample from the roof monitor to ground level. A typical installation of duct for drawing a sample from a roof monitor to ground level is shown in Figure 14-1. A plan of a manifold system that is located in a roof monitor is shown in Figure 14-2. These drawings represent a typical installation for a generalized roof monitor. The dimensions on these figures may be altered slightly to make the manifold system fit into a particular roof monitor, but the general configuration shall be followed. There shall be eight nozzles, each having a diameter of 0.40 to 0.50 meters. The length of the manifold system from the first nozzle to the eighth shall be 35 meters or eight percent of the length of the roof monitor, whichever is greater. The duct leading from the roof monitor manifold shall be round with a diameter of 0.30 to 0.40 meters. As shown in Figure 14-2, each of the sample legs of the manifold shall have a device, such as a blast gate or valve, to enable adjustment of flow into each sample nozzle.

Locate the manifold along the length of the roof monitor so that it lies near the midsection of the roof monitor. If the design of a particular roof monitor makes this impossible, the manifold may be located elsewhere along the roof monitor, but avoid locating the manifold near the ends of the roof monitor or in a section where the aluminum reduction pot arrangement is not typical of the rest of the potroom. Center the sample nozzles in the throat of the roof monitor. (See Figure 14-1.) Construct all

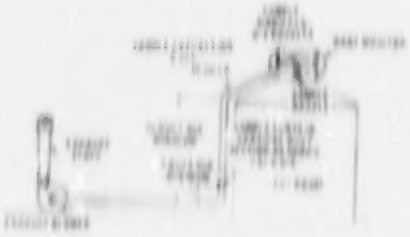

Figure 14-1. Roof monitor sampling system.

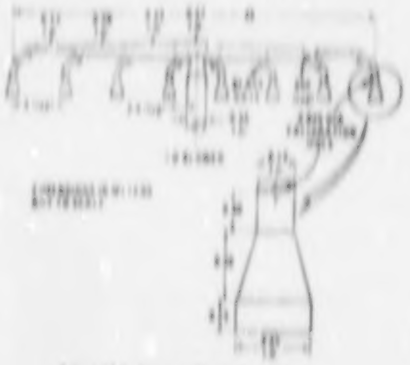

Figure 14-2. Sampling manifold and nozzles.

sample-exposed surfaces within the sampling manifold and sample duct of 316 stainless steel. Aluminum may be used if a new ductwork system is conditioned with fluoride-laden roof monitor air for a period of six weeks prior to initial testing. Other materials of construction may be used if it is demonstrated through comparative testing that there is no loss of fluorides in the system. All connections in the ductwork shall be leak free.

Locate two sample ports in a vertical section of the duct between the roof monitor and exhaust fan. The sample ports shall be at least 10 duct diameters downstream and two diameters upstream from any flow disturbance such as a bend or contraction. The two sample ports shall be situated 90° apart. One of the sample ports shall be situated so that the duct can be traversed in the plane of the nearest upstream duct bend.

2.2.3 *Exhaust fan.* An industrial fan or blower to be attached to the sample duct at ground level. (See Figure 14-1.) This exhaust fan shall have a maximum capacity such that a large enough volume of air can be pulled through the ductwork to maintain an isokinetic sampling rate in all the sample nozzles for all flow rates normally encountered in the roof monitor.

The exhaust fan volumetric flow rate shall be adjustable so that the roof monitor air can be drawn isokinetically into the sample nozzles. This control of flow may be achieved by a damper on the inlet to the exhauster or by any other workable method.

2.3 *Temperature measurement apparatus.*

2.3.1 *Thermocouple.* Installed in the roof monitor near the sample duct.

2.3.2 *Signal transducer.* Transducer to change the thermocouple voltage output to a temperature readout.

2.3.3 *Thermocouple wire.* To reach from roof monitor to signal transducer and recorder.

2.4 *Sampling train.* Use the train described in Methods 13A and 13B—Determination of total fluoride emissions from stationary sources.

3. *Reagents*

3.1 *Sampling and analysis.* Use reagents described in Method 13A or 13B—Determination of total fluoride emissions from stationary sources.

4. *Calibration*

4.1 *Propeller anemometer.* Calibrate the anemometers so that their electrical signal output corresponds to the velocity or volumetric flow they are measuring. Calibrate according to manufacturer's instructions.

4.2 *Manifold intake nozzles.* Adjust the exhaust fan to draw a volumetric flow rate (refer to Equation 14-1) such that the entrance velocity into each manifold nozzle approximates the average effluent velocity in the roof monitor. Measure the velocity of the air entering each nozzle by inserting an S type pitot tube into a 2.5 cm or less diameter hole (see Figure 14-2) located in the manifold between each blast gate (or valve) and nozzle. The pitot tube tip shall be extended into the center of the manifold. Take care to insure that there is no leakage around the pitot probe which could affect the indicated velocity in the manifold leg. If the velocity of air being drawn into each nozzle is not the same, open or close each blast gate (or valve) until the velocity in each nozzle is the same. Fasten each blast gate (or valve) so that it will remain in this position and close the pitot port holes. This calibration shall be performed when the manifold system is installed. (Note: It is recommended that this calibration be repeated at least once a year.)

5. *Procedure*

5.1 *Roof monitor velocity determination.*

5.1.1 *Velocity value for setting isokinetic flow.* During the 24 hours preceding a test run, determine the velocity indicated by the propeller anemometer in the section of roof monitor containing the sampling manifold. Velocity readings shall be taken every 15 minutes or at shorter equal time intervals. Calculate the average velocity for the 24-hour period.

5.1.2 *Velocity determination during a test run.* During the actual test run, record the velocity or volume readings of each propeller anemometer in the roof monitor. Velocity readings shall be taken for each anemometer every 15 minutes or at shorter equal time intervals (or continuously).

5.2 *Temperature recording.* Record the temperature of the roof monitor every two hours during the test run.

5.3 *Sampling.*

5.3.1 *Preliminary air flow in duct.* During the 24 hours preceding the test, turn on the exhaust fan and draw roof monitor air through the manifold duct to condition the ductwork. Adjust the fan to draw a volumetric flow through the duct such that the velocity of gas entering the manifold nozzles approximates the average velocity of the air exiting the roof monitor.

5.3.2 *Isokinetic sample rate adjustment.* Adjust the fan so that the volumetric flow rate in the duct is such that air enters into the manifold sample nozzles at a velocity equal to the 24-hour average velocity determined under 5.1.1. Equation 14-1 gives the correct stream velocity which is needed in the duct at the sample ports in order for sample gas to be drawn isokinetically into the manifold nozzles. Perform a pitot traverse of the duct at the sample ports to determine if the correct average velocity in the duct has been achieved. Perform the pitot determination according to Method 2. Make this determination before the start of a test run. The fan setting need not be changed during the run.

$$V_d = \frac{8\,(D_n)^2}{(D_d)^2}\,(V_m)\,\frac{1\ \text{minute}}{60\ \text{sec}} \qquad \text{Eq. 14-1}$$

where:

$V_d$=desired velocity in duct at sample ports, meter/sec.

$D_n$=diameter of a roof monitor manifold nozzle, meters.

$D_d$=diameter of duct at sample port, meters.

$V_m$=average velocity of the air stream in the roof monitor, meters/minute, as determined under section 5.1.1.

5.3.3 *Sample train operation.* Sample the duct using the standard fluoride train and methods described in Methods 13A and 13B—Determination of total fluoride emissions from stationary sources. Select sample traverse points according to Method 1. If a selected sampling point is less than 1 inch from the stack wall, adjust the location of that point to one inch away from the wall.

5.3.4 Each test run shall last eight hours or more. If a question exists concerning the representativeness of an eight-hour test, a longer test period up to 24 hours may be selected. Conduct each run during a period when all normal operations are performed underneath the sampling manifold, i.e. tapping, anode changes, maintenance, and other normal duties. All pots in the potroom shall be operated in a normal manner during the test period.

5.4 *Sample recovery.* Same as Method 13A or 13B—Determination of total fluoride emissions from stationary sources.

5.5 *Analysis.* Same as Method 13A or 13B—Determination of total fluoride emissions from stationary sources.

8. *Calculations*

8.1 *Isokinetic sampling test.* Calculate the mean velocity measured during each sampling run by the anemometer in the section of the roof monitor containing the sampling manifold. If the mean velocity recorded during a particular test run does not fall within ±10 percent of the mean velocity established according to 6.2.3, repeat the run.

8.2 *Average velocity of roof monitor gases.* Calculate the average roof monitor velocity using all the velocity or volumetric flow readings from section 6.1.2.

8.3 *Roof monitor temperature.* Calculate the mean value of the temperatures recorded in section 6.2.

8.4 *Concentration of fluorides in roof monitor air in mg F/m³.* This is given by Equation 13A-5 in Method 13A—Determination of total fluoride emissions from stationary sources.

8.5 *Average volumetric flow from roof* is given by Equation 14-2.

$$Q_m = \frac{V_m (A) (M_d) P_m (293°K)}{(T_m + 273°) (760 \text{ mm Hg})} \quad \text{Eq. 14-2}$$

where:

$Q_m$ = average volumetric flow from roof monitor at standard conditions on a dry basis, m³/min.

$A$ = roof monitor open area, m².

$V_m$ = average velocity of air in the roof monitor, meters/minute, from section 8.2.

$P_m$ = atmospheric pressure, mm Hg.

$T_m$ = roof monitor temperature, °C, from section 8.3.

$M_d$ = mole fraction of dry gas, which is given by $M_d = \frac{100 - 100 (B_{wo})}{100}$

$B_{wo}$ = is the proportion by volume of water vapor in the gas stream, from Equation 13A-3, Method 13A—Determination of total fluoride emissions from stationary sources.

[36 FR 24877, Dec. 23, 1971, as amended at 38 FR 13562, May 23, 1973; 39 FR 9319, Mar. 8, 1974; 39 FR 13776, Apr. 17, 1974; 39 FR 20794, June 14, 1974; 39 FR 39873, Nov. 12, 1974; 40 FR 33157, Aug. 6, 1975; 41 FR 3826, Jan. 26, 1976]

# APPENDIX H

## FEDERAL STATIONARY SOURCE PERFORMANCE STANDARDS

Since December 1971 the Environmental Protection Agency has promulgated source performance standards for twenty industrial air pollution sources. These regulations have been presented in numerous issues of the Federal Register and can now be found in the Code of Federal Regulations under Title 40 - Protection of Environment; Chapter 1 - Environmental Protection Agency; Subchapter C - Air Programs; Part 60 - Standards of Performance For New Stationary Sources.

This appendix presents a summary of these regulations as found in the Code of Federal Regulations. This summary is an updated listing as found originally in the following reference:

L. S. Chaput. 1976. Federal Standards of Performance For New Stationary Sources Of Air Pollution - A Summary of Regulations. Journal of the Air Pollution Control Association, Vol. 26, No. 11; 1055-1060.

| Source category | Affected facility | Pollutant | Emission level | Monitoring requirement |
|---|---|---|---|---|
| **Subpart D:** | | | | |
| **Steam generators**<br>(>250 million Btu/hr)<br>*Promulgated*<br>12/23/71 (36 FR 24876)<br>*Revised*<br>7/26/72 (37 FR 14877)<br>10/15/73 (38 FR 28565)<br>6/14/74 (39 FR 20790)<br>1/16/75 (40 FR 2803)<br>10/6/75 (40 FR 46250)<br>11/22/76 (41 FR 51398)<br>1/31/77 (42 FR 5936) | Coal fired boilers | Particulate<br>Opacity<br>$SO_2$<br>$NO_x$ (except lignite and coal refuse) | 0.10 lb/$10^6$ Btu<br>20%<br>1.2 lb/$10^6$ Btu<br>0.70 lb/$10^6$ Btu | No requirement<br>Continuous<br>Continuous<br>Continuous |
| | Oil fired boilers | Particulate<br>Opacity<br>$SO_2$<br>$NO_x$ | 0.10 lb/$10^6$ Btu<br>20%; 40% 2 min/hr<br>0.80 lb/$10^6$ Btu<br>0.30 lb/$10^6$ Btu | No requirement<br>Continuous<br>Continuous<br>Continuous |
| | Gas fired boilers | Particulate<br>Opacity<br>$NO_x$ | 0.10 lb/$10^6$ Btu<br>20%<br>0.20 lb/$10^6$ Btu | No requirement<br>No requirement<br>Continuous |
| **Subpart E:** | | | | |
| **Incinerators**<br>(>50 tons/day)<br>*Promulgated*<br>12/23/71 (36 FR 24876)<br>*Revised*<br>6/14/74 (39 FR 20790) | Incinerators | Particulate | 0.08 gr/dscf corrected to 12% $CO_2$ | No requirement |
| **Subpart F:** | | | | |
| **Portland cement plants**<br>*Promulgated*<br>12/23/71 (36 FR 24876)<br>*Revised*<br>6/14/74 (39 FR 20790)<br>11/12/74 (39 FR 39874)<br>10/6/75 (40 FR 46250) | Kiln | Particulate<br>Opacity | 0.30 lb/ton<br>20% | No requirement<br>No requirement |
| | Clinker cooler | Particulate<br>Opacity | 0.10 lb/ton<br>10% | No requirement<br>No requirement |
| | Fugitive Emission points | Opacity | 10% | No requirement |
| **Subpart G:** | | | | |
| **Nitric acid plants**<br>*Promulgated*<br>12/23/71 (36 FR 2[illegible]76)<br>*Revised*<br>5/23/73 (38 FR 13562)<br>10/15/73 (38 FR 28565)<br>6/14/74 (39 FR 20790)<br>10/6/75 (40 FR 46250) | Process equipment | Opacity<br>$NO_x$ | 10%<br>3.0 lb/ton | No requirement<br>Continuous |
| **Subpart H:** | | | | |
| **Sulfuric acid plants**<br>*Promulgated*<br>12/23/71 (36 FR 24876)<br>*Revised*<br>5/23/73 (38 FR 13562)<br>10/15/73 (38 FR 28565)<br>6/14/74 (39 FR 20790)<br>10/6/75 (40 FR 46250) | Process equipment | $SO_2$<br>Acid mist<br>Opacity | 4.0 lb/ton<br>0.15 lb/ton<br>10% | Continuous<br>No requirement<br>No requirement |

| Source category | Affected facility | Pollutant | Emission level | Monitoring requirement |
|---|---|---|---|---|
| **Subpart I:** | | | | |
| **Asphalt concrete plants** | Dryers; screening and weighing systems; storage, transfer, and loading systems; and dust handling equipment | Particulate | 0.04 gr/dscf (90 mg/dscm) | No requirement |
| *Promulgated*<br>3/8/74 (39 FR 9308) | | Opacity | 20% | No requirement |
| *Revised*<br>10/6/75 (40 FR 46250) | | | | |
| **Subpart J:** | | | | |
| **Petroleum refineries** | Catalytic cracker | Particulate<br>Opacity<br>CO | 1.0 lb/1000 lb<br>30% (3 min. exemption)<br>0.05% | No requirement<br>Continuous<br>Continuous |
| *Promulgated*<br>3/8/74 (39 FR 9308) | | | | |
| *Revised*<br>10/6/75 (40 FR 46250) | Fuel gas combustion | SO$_2$ | 0.1 gr $H_2S$/dscf (230 mg/dscm) | Continuous |
| **Subpart K:** | | | | |
| **Storage vessels for petroleum liquids** | Storage tanks $>$40,000 gal. capacity | Hydrocarbons | For vapor pressure 78-570 mm Hg, equip with floating roof, vapor recovery system, or equivalent; for vapor pressure $>$570 mm Hg, equip with vapor recovery system or equivalent | No requirement |
| *Promulgated*<br>3/8/74 (39 FR 9308) | | | | |
| *Revised*<br>4/17/74 (39 FR 13776)<br>6/14/74 (39 FR 20790) | | | | |
| **Subpart L:** | | | | |
| **Secondary lead smelters** | Reverberatory and blast furnaces | Particulate | 0.022 gr/dscf (50 mg/dscm) | No requirement |
| *Promulgated*<br>3/8/74 (39 FR 9308) | | Opacity | 20% | No requirement |
| | Pot furnaces | Opacity | 10% | No requirement |
| *Revised*<br>4/17/74 (39 FR 13776)<br>10/6/75 (40 FR 46250) | | | | |
| **Subpart M:** | | | | |
| **Secondary brass and bronze plants** | Reverberatory furnace | Particulate | 0.022 gr/dscf (50 mg/dscm) | No requirement |
| *Promulgated*<br>3/8/74 (39 FR 9308) | | Opacity | 20% | No requirement |
| *Revised*<br>10/6/75 (40 FR 46250) | Blast and electric furnaces | Opacity | 10% | No requirement |
| **Subpart N:** | | | | |
| **Iron and steel plants** | Basic oxygen process furnace | Particulate | 0.022 gr/dscf (50 mg/dscm) | No requirement |
| *Promulgated*<br>3/8/74 (39 FR 9308) | | | | |

| Source category | Affected facility | Pollutant | Emission level | Monitoring requirement |
|---|---|---|---|---|
| **Subpart O:** | | | | |
| **Sewage treatment plants**<br>*Promulgated*<br>3/8/74 (39 FR 9308)<br>*Revised*<br>4/17/74 (39 FR 13776)<br>5/3/74 (39 FR 15396)<br>10/6/75 (40 FR 46250) | Sludge incinerators | Particulate<br>Opacity | 1.30 lb/ton<br>20% | Mass or volume of sludge<br>No requirement |
| **Subpart P:** | | | | |
| **Primary copper smelters**<br>*Promulgated*<br>1/15/76 (41 FR 2331)<br>*Revised*<br>2/26/76 (41 FR 8346) | Dryer | Particulate<br>Opacity | 0.022 gr/dscf (50 mg/dscm)<br>20% | No requirement<br>Continuous |
| | Roaster, smelting furnace,[1] copper converter<br>[1] Reverberatory furnaces that process high-impurity feed materials are exempt from $SO_2$ standard | $SO_2$<br>Opacity | 0.065%<br>20% | Continuous<br>No requirement |
| **Subpart Q:** | | | | |
| **Primary zinc smelters**<br>*Promulgated*<br>1/15/76 (41 FR 2331) | Sintering machine | Particulate<br>Opacity | 0.022 gr/dscf (50 mg/dscm)<br>20% | No requirement<br>Continuous |
| | Roaster | $SO_2$<br>Opacity | 0.065%<br>20% | Continuous<br>No requirement |
| **Subpart R:** | | | | |
| **Primary lead smelters**<br>*Promulgated*<br>1/15/76 (41 FR 2331) | Blast or reverberatory furnace, sintering machine discharge end | Particulate<br>Opacity | 0.022 gr/dscf (50 mg/dscm)<br>20% | No requirement<br>Continuous |
| | Sintering machine electric smelting furnace, converter | $SO_2$<br>Opacity | 0.065%<br>20% | Continuous<br>No requirement |
| **Subpart S:** | | | | |
| **Primary aluminum reduction plants**<br>*Promulgated*<br>1/26/76 (41 FR 3825) | Potroom group<br>(a) Soderberg plant<br>(b) Prebake plant | (a) Total fluorides<br>Opacity<br>(b) Total fluorides<br>Opacity | 2.0 lb/ton<br>10%<br>1.9 lb/ton<br>10% | No requirement<br>No requirement<br>No requirement<br>No requirement |
| | Anode bake plants | Total fluorides<br>Opacity | 0.1 lb/ton<br>20% | No requirement<br>No requirement |

| Source category | Affected facility | Pollutant | Emission level | Monitoring requirement |
|---|---|---|---|---|
| **Subpart T:** | | | | |
| **Phosphate fertilizer plants**<br>*Promulgated*<br>8/6/75 (40 FR 33152) | Wet process phosphoric acid | Total fluorides | 0.02 lb/ton | Total pressure drop across process scrubbing system |
| **Subpart U:** | Superphosphoric acid | Total fluorides | 0.01 lb/ton | Total pressure drop across process scrubbing system |
| **Subpart V:** | Diammonium phosphate | Total fluorides | 0.06 lb/ton | Total pressure drop across process scrubbing system |
| **Subpart W:** | Triple superphosphate | Total fluorides | 0.2 lb/ton | Total pressure drop across process scrubbing system |
| **Subpart X:** | Granular triple superphosphate | Total fluorides | $5.0 \times 10^{-4}$ lb/hr/ton | Total pressure drop across process scrubbing system |
| **Subpart Y:** | | | | |
| **Coal preparation plants**<br>*Promulgated*<br>1/15/76 (41 FR 2232) | Thermal dryer | Particulate | 0.031 gr/dscf (0.070 g/dscm) | Temperature<br>Scrubber pressure loss<br>Water pressure |
| | | Opacity | 20% | No requirement |
| | Pneumatic coal cleaning equipment | Particulate | 0.018 gr/dscf (0.040 g/dscm) | No requirement |
| | | Opacity | 10% | No requirement |
| | Processing and Conveying equipment, storage systems, transfer and loading systems | Opacity | 20% | No requirement |
| **Subpart Z:** | | | | |
| **Ferroalloy production facilities**<br>*Promulgated*<br>5/4/76 (41 FR 18497)<br>*Revised*<br>5/20/76 (41 FR 20659) | Electric submerged arc furnaces | Particulate | 0.99 lb/Mw-hr (0.45 kg/Mw-hr) ("high silicon alloys")<br>0.51 lb/Mw-hr (0.23 kg/Mw-hr) (chrome and manganese alloys) | No requirement |
| | | | No visible emissions may escape furnace capture system | Flowrate monitoring in hood |

| Source category | Affected facility | Pollutant | Emission level | Monitoring requirement |
|---|---|---|---|---|
| **Ferroalloy production facilities (cont.)** | | | No visible emission may escape tapping system for >40% of each tapping period | Flowrate monitoring in hood |
| | | Opacity<br>CO | 15%<br>20% volume basis | Continuous<br>No requirement |
| | Dust handing equipment | Opacity | 10% | No requirement |
| **Subpart AA:** | | | | |
| **Iron and steel plants**<br>*Promulgated*<br>9/23/75 (40 FR 43850) | Electric arc furnaces | Particulate | 0.0052 gr/dscf<br>(12 mg/dscm) | No requirement |
| | | Opacity<br>(a) control device | 3% | Continuous |
| | | (b) shop roof | 0, except<br>20%—charging<br>40%—tapping | Flowrate monitoring in capture hood<br>Pressure monitoring in DSE system |
| | Dust handing equipment | Opacity | 10% | No requirement |

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